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ENGINEERING EXPERIMENT STATION

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PROJECT INITIATION

Date: April 4, 1975

Project Title: An Investigation of Reaction Sintered Nitride as a Radome Material

Project No.: A-1724

Project Director: Mr. Joe N. Harris

Sponsor: Naval Air Systems Command; Washington, D. C. 20360

Agreement Period: From 3/1/75 Until 8/31/75

Type Agreement: Contract No. N00019-75-C-0372

Amount: $20,731

Reports Required: Monthly Progress Letters; Final Technical Report

Sponsor Contact Person:

Technical Matters
Naval Air Systems Command
Code: 52032
Washington, D. C. 20360

Contractual Matters
(Thru GTRI)
Mr. R. J. Whitcomb (ACO)
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Defense Priority Rating: DO-C9 under DMS Reg. 1

Assigned to: HIGH TEMPERATURE MATERIALS DIVISION

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PROJECT TERMINATION

Date: February 4, 1976

Project Title: An Investigation of Reaction Sintered Nitride as a Radome Material

Project No: A-1724

Project Director: Mr. J. N. Harris

Sponsor: Naval Air Systems Command

Effective Termination Date: 11/30/75

Clearance of Accounting Charges: 1/31/76

Grant/Contract Closeout Actions Remaining:

1. Final Report of Inventions
2. Final Invoice
3. Gov't Property Inven. & Cert.
4. Classified Material Certificate

Assigned to: Applied Sciences Laboratory

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orm EES 402 (R10–75)

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

April 7, 1975

Department of the Navy
Naval Air Systems Command
Washington, D. C. 20360

Code: AIR-52032

Subject: "An Investigation of Reaction Sintered Silicon Nitride as a Radome Material," Progress Report No. 1, Contract N00019-75-C-0372 for the Period 1 March-31 March 1975, Project A-1724

Gentlemen:

The work in progress under Contract N00019-74-C-0248 was continued. Two "Mauler" radomes were precision slip-cast from silicon slip to which 2½ weight percent pigment grade magnitite (black iron oxide, $FeO \cdot Fe_2O_3$) had been added. The first radome was cast from a 75 percent solids slip with a viscosity of 788 centipoise* and a pH of 6.9. This thick slip was used in an attempt to produce a lower density casting. The casting produced was much lighter than those produced using lower viscosity (< 200 centipoise) slip, but did not cast evenly. The surface was covered with "dimples" caused by too rapid casting with subsequent "bridging" over in some areas. As drying progressed the "bridged" areas shrank inwardly producing the dimpled surface. Even though this radome had imperfections it was still reaction sintered. Weight gain from reaction sintering was 61 percent, therefore, this radome was submitted for measurement of transmission efficiency in the area not affected by the dimples. This data should be available for inclusion in the next letter report.

The high viscosity slip was modified to lower the viscosity to 665 centipoise in an effort to eliminate the dimpling problem. A casting made with this slip produced a radome with only one imperfection. However, upon drying this radome cracked with the crack emanating from the imperfection. Future attempts at casting radome shapes will be limited to slips with viscosities not exceeding 350 centipoise.

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*As measured with a Brookfield Viscometer Model LV, 30 rpm, No. 2 spindle.

One other radome was reaction sintered during this report period. It was a high density casting which only increased in weight by 53 percent. Therefore, it was not checked for electrical transmission.

Two large closed end alumina tubes used as retorts for reaction sintering Sparrow size radomes both cracked due to thermal shock. Therefore, another approach is being taken to sinter these shapes. Slip-cast fused silica retorts will be used for single firings only since they are expected to devitrify at the upper reaction sintering temperature (2600° F). A Hawk radome model is being used to prepare molds for slip casting fused silica retorts.

The Sparrow tooling used to slip-cast silicon radome blanks under Contract N00019-74-C-0248 belonged to another program and is no longer available for use. However, this tooling was designed for fused silica and produced a blank considerably thicker (0.470 inch) than desired for silicon nitride. An internal mandrel is to be prepared for use with an existing Sparrow master model to produce slip-cast silicon blanks 0.270 inch thick. This will make nitriding of the blank easier and will reduce considerably any requirements for machining a radome to be boresighted.

During the next report period the above tooling will be finished and data will be available on the effects of iron oxide additions to the silicon slip on electrical transmission properties of reaction sintered silicon nitride radomes. If the iron oxide in quantities up to 2½ percent is not detrimental to the electrical properties, then additions of 0 to 2½ percent iron oxide will be used to adjust the rheological properties of slips for casting radome blanks. This will provide enhanced reaction sintering and a method of controlling the cast density of the parts. Both Mauler and Sparrow size radomes will be cast and reaction sintered.

Respectfully submitted,

Joe N. Harris
Project Director

jw

A-1724

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

May 26, 1975

Department of the Navy
Naval Air Systems Command
Washington, D. C. 20360

Code: AIR-52032

Subject: "An Investigation of Reaction Sintered Silicon Nitride as a Radome Material," Progress Report No. 2, Contract N00019-75-C-0372 for the Period 1 April-30 April 1975, Project A-1724

Gentlemen:

An additional one hundred pounds of Union Carbide MXD-325 mesh silicon powder was dry milled for 17 hours in high alumina ball mills with high density alumina grinding media. This material will be used to make additional aqueous silicon slip. Two additional "Mauler" radomes were precision slip-cast, dried and reaction sintered. The first radome was made from a silicon slip, 65 days old, containing one percent added iron oxide. The total solids content was 75 percent. Viscosity was 80 centipoise and pH was 6.45. This radome cast to an estimated green density of 1.66 gm/cm^3. This radome was nitrided using a "standard firing cycle" of 24 hours at 2000^o F, 24 hours at 2380^o F and 24 hours at 2600^o F. After cooling this radome was found to have only gained 55 percent weight.

The second radome was cast from an 80 percent solids slip not containing any added iron oxide. This slip was 66 days in age, had a pH of 5.8 and a viscosity of 420 centipoise. The higher viscosity was expected to produce a lower density casting, however, the estimated green density of this casting was 1.80 gm/cm^3. An attempt was made to increase the degree of nitridation of this denser casting by increasing the nitriding time at 2380^o F. An additional 24 hours was added to the "standard" firing cycle, however, percent weight pick-up after nitriding was only 46.1.

Dr. Don Messier of the Army Materials and Mechanics Research Center nitrided a 2 inch diameter by 1/4 inch thick disc slip-cast at Georgia Tech. He was able to achieve a 62.8 percent weight gain in this disc. Final density was 2.67 gm/cm^3. X-ray analysis indicated that this disc was 36 percent α and 64 percent β silicon nitride. No silicon was detected. Dr. Messier achieved this degree of nitridation by using the following schedule:

3 hours at 2120° F
24 hours at 2318° F
12 hours at 2408° F
4 hours at 2534° F
24 hours at 2552° F

During the next report period the above schedule will be used on Mauler radomes in an effort to achieve more complete nitridation.

A mold was made to slip-cast a fused silica retort to be used in nitriding Sparrow sized radomes. During the next report period the retort will be cast and Sparrow size radomes will be nitrided.

Respectfully submitted,

Joe N. Harris
Project Director

jw

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

August 4, 1975

Department of the Navy
Naval Air Systems Command
Washington, D. C. 20360

Code Air - 52032

Subject: "An Investigation of Reaction Sintered Silicon Nitride as a Radome Material," Progress Report No. 4, Contract N00019-75-C-0372 for the Period, 1 June - 30 June 1975, Project A-1724

Gentlemen:

Additional silicon slip containing 2.75 weight percent magnetite was prepared. This slip was used to cast two Mauler size radome blanks. These blanks were cast four days apart and were numbers eleven and twelve in the current series of radome blanks.

Prior experience has demonstrated that during nitridation a severe exothermic reaction begins when the thermocouple inside the retort reaches 2060°F. This thermocouple is enclosed in an alumina protection tube, therefore, the temperature inside the retort is probably somewhat higher.

Due to starting the temperature program too early on the previous day radome number 11 reached an indicated 2060°F about seven A.M. By the time someone noticed this the chamber pressure had just gone negative. Argon was added to the system to obtain a positive pressure and slow down the reaction rate but, apparently sufficient air had already entered the chamber to oxidize the surface of the unnitrided silicon. Weight pick-up after nitridation was only 48%. The start-up program was lengthened 4 hours and radome 12 was nitrided. The pressure in the system was observed closely as the temperature approached 2060°F and argon was added to maintain a pressure in the retort of 7.5 inches of water. This operation is conducted manually. As soon as the reaction slows the argon is shut-off. After nitridation radome 12 had a weight increase of more than 59 percent.

A master model was prepared from aluminum to form a cylindrical dense silicon casting with internal 8-pitch threads. The purpose of this part will be to demonstrate the capability of bonding a dense reaction sintered silicon nitride part to a more porous radome. Some initial castings were made and these will be sintered during July.

The Hawk radome plaster mold intended for use in preparing a slip cast fused silica retort to nitride a sparrow size radome was modified to produce an 8 inch diameter rounded tip by filling the tip of the mold with plaster. The fused silica retort will be slip-cast and bisque fired in July and used to nitride a sparrow size radome.

Respectfully Submitted,

J. N. Harris
Project Director

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

August 4, 1975

Department of the Navy
Naval Air Systems Command
Washington, D. C. 20360

Code: AIR-52032

Subject: "An Investigation of Reaction Sintered Silicon Nitride as a Radome Material," Progress Report No. 5, Contract N00019-75-C-0372 for the Period, 1 July-31 July 1975, Project A-1724

Gentlemen:

Three additional Mauler radome blanks (numbers 13, 14 and 15) were cast during this period. An attempt was made to cast an additional Sparrow size radome, but leaks in the casting set-up precluded the use of pressure. The Sparrow blank was cast without pressure, but this resulted in shrinkage against the internal mandrel and cracking.

Several threaded rings were cast from a silicon slip which produces a dense silicon casting. Two of these rings were argon sintered to provide sufficient strength to accomplish sanding and clean up operations on the outer surface. One of the argon sintered rings was fitted into the base of the dry Mauler radome blank number 15. A low viscosity silicon slip was used to fill the area between the argon sintered casting and the dry radome. This radome and the attachment ring were reaction sintered in nitrogen. After sintering they were essentially one piece and it was not possible to see the joint between radome and attachment ring. This radome is shown in Figure 1.

A fused silica retort was slip-cast and sintered to be used in reaction sintering a Sparrow radome. The as-cast retort is shown in Figure 2. This retort will allow the reaction sintering of one Sparrow radome during the final month of the program. If this radome is successfully reaction sintered it will be fitted with an Invar attachment ring and provided to the Air Force Materials laboratory for future sled testing. If this radome is not successfully fired, then one of the smaller Mauler shapes will be fitted with a metal ring suitable for sled testing and this will be provided to the Air Force Materials laboratory.

Four of the reaction sintered Mauler radomes were checked for electrical transmission characteristics by placing an antenna inside the radome and transmitting through the wall over the frequency range of 8 to 12 GHz. Patterns were made on four dry radomes and on three of the four radomes after

they had been sitting in very humid conditions for several days. Only one of the radomes showed any severe pattern degradation due to the presence of moisture. Patterns for each of the radomes are attached to this report. The cross-hatched pattern line is transmission through the radome.

It had previously been assumed that the best transmission would be obtained in a radome which had the highest percentage of theoretical weight increase after nitridation. This assumption was based on previous pattern measurements on radome number 2 which had an apparent weight increase during nitridation of 62.4 percent. A recheck of the transmission of radome number 2 showed that it was not as good as radome number 3 which had an apparent increase of only 49.9 percent after nitridation.

During the final report period radome number 3 will be sealed by impregnating with G.E. SR-80 silicone resin. Transmission measurements will then be made with the radome thoroughly dry and after immersing the radome in water then drying the outer surface. The final report draft will be completed and submitted for approval.

Respectfully submitted,

Joe N. Harris
Project Director

jw

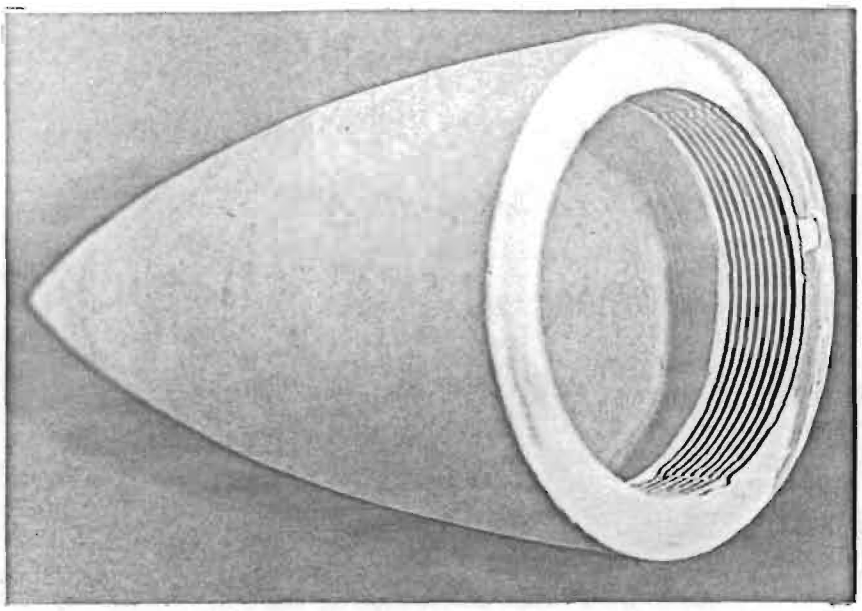

Figure 1. Reaction Sintered Silicon Nitride (RSSN) Radome and RSSN Attachment Ring.

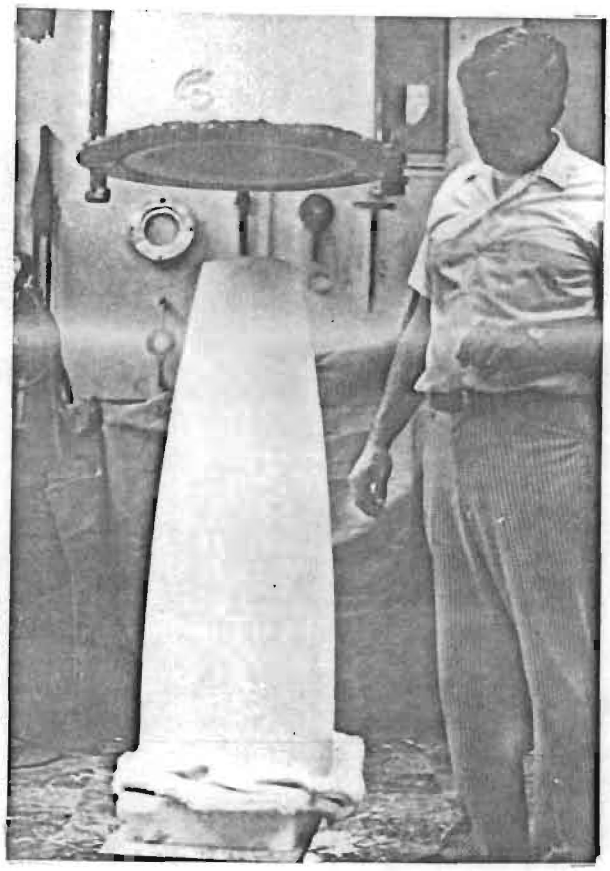

Figure 2. Fused Silica Retort for Reaction Sintering Sparrow Size Radome.

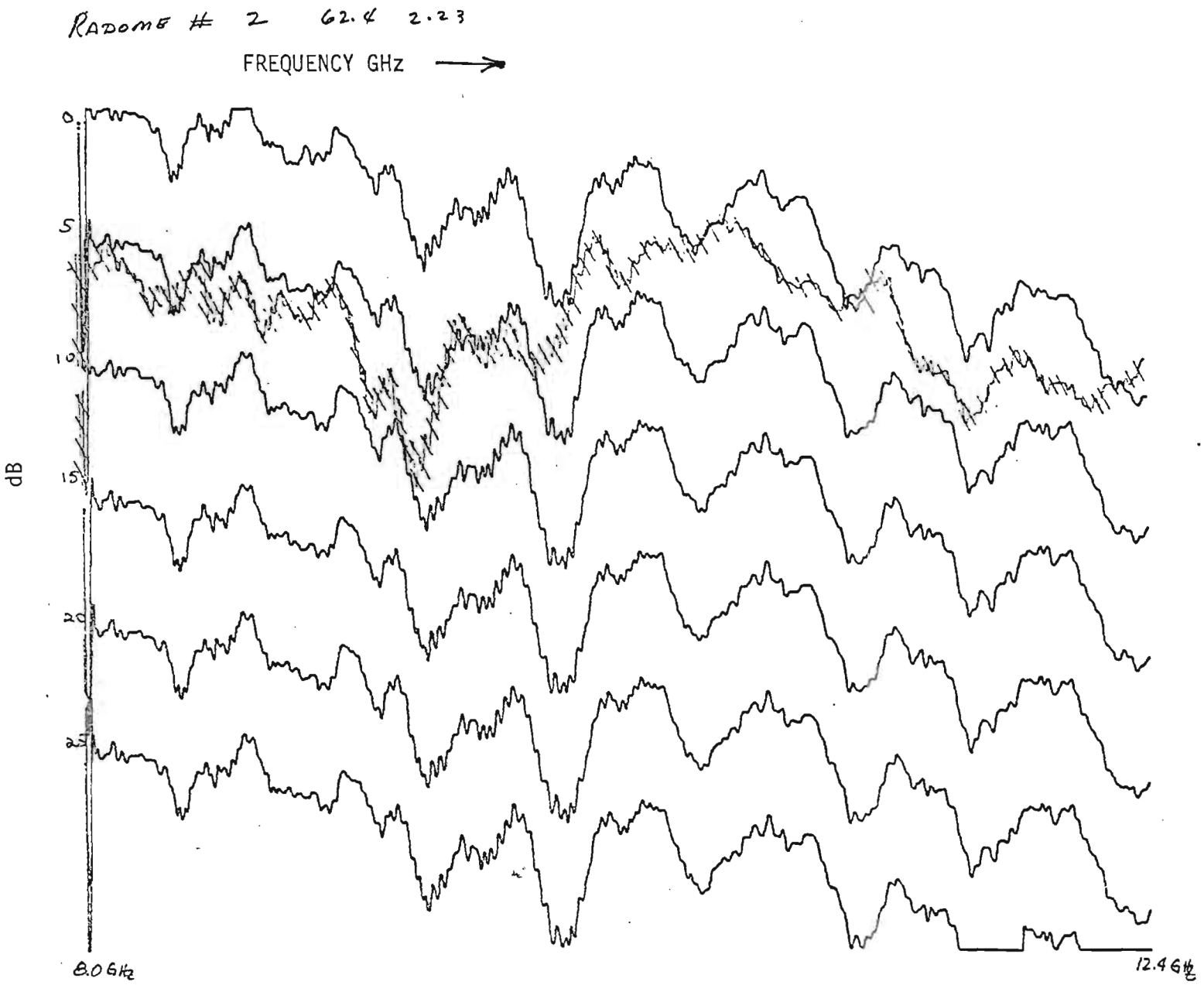

Figure 3. Transmission Through Radome Number 2 (After Thorough Drying).

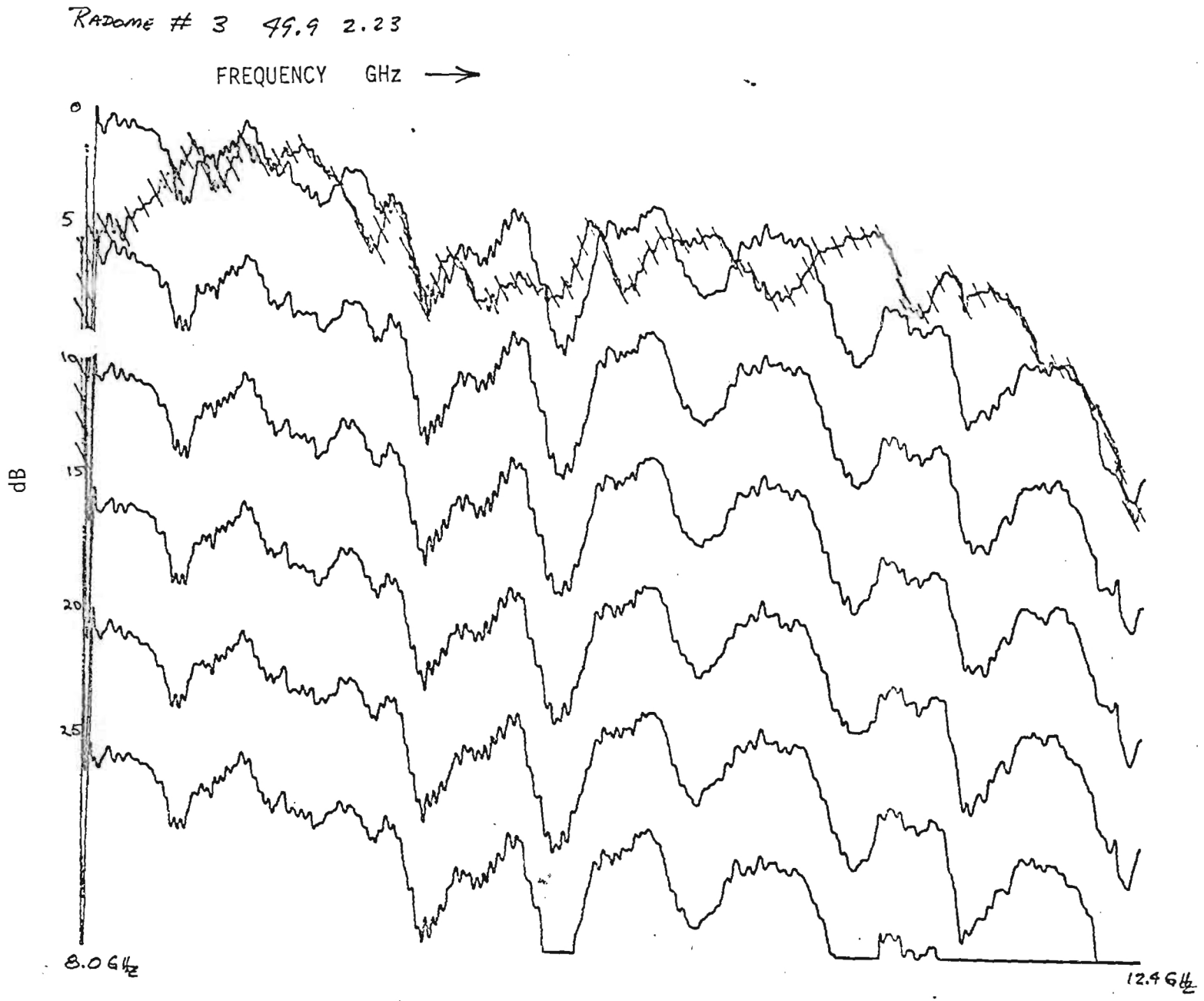

Figure 4. Transmission Through Radome Number 3 (After Thorough Drying).

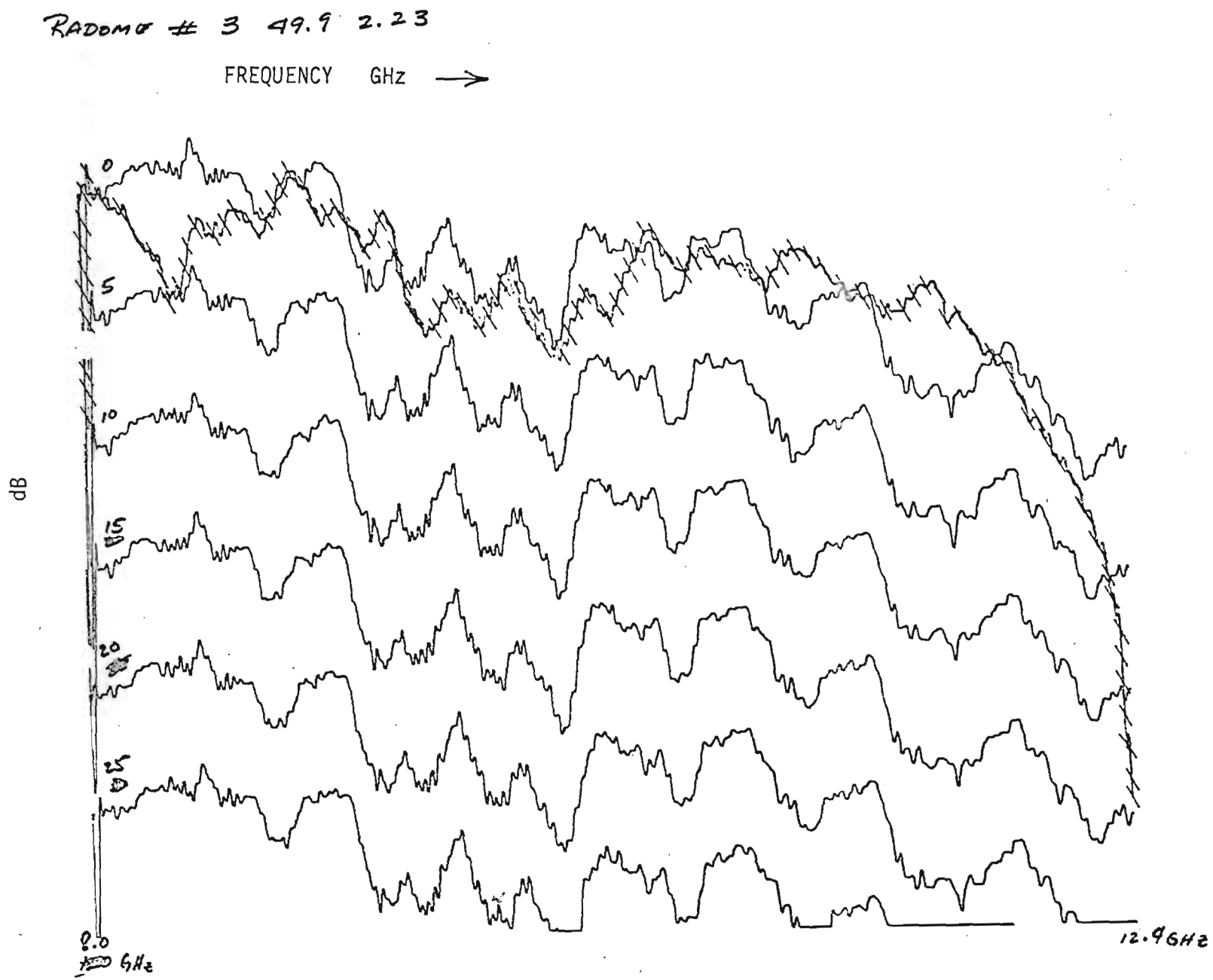

Figure 5. Transmission Through Radome Number 3 (After Exposure to Laboratory Humidity).

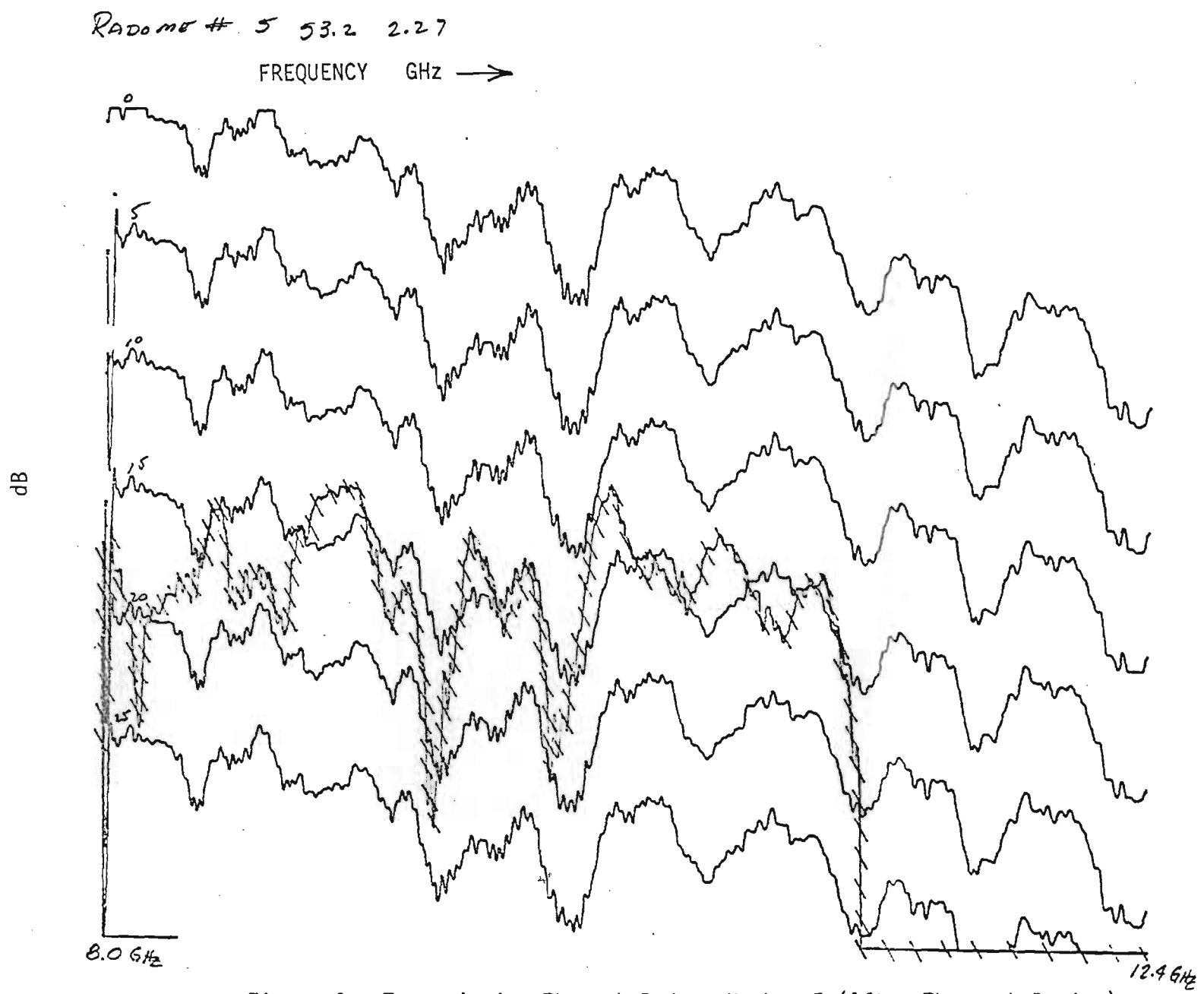

Figure 6. Transmission Through Radome Number 5 (After Thorough Drying).

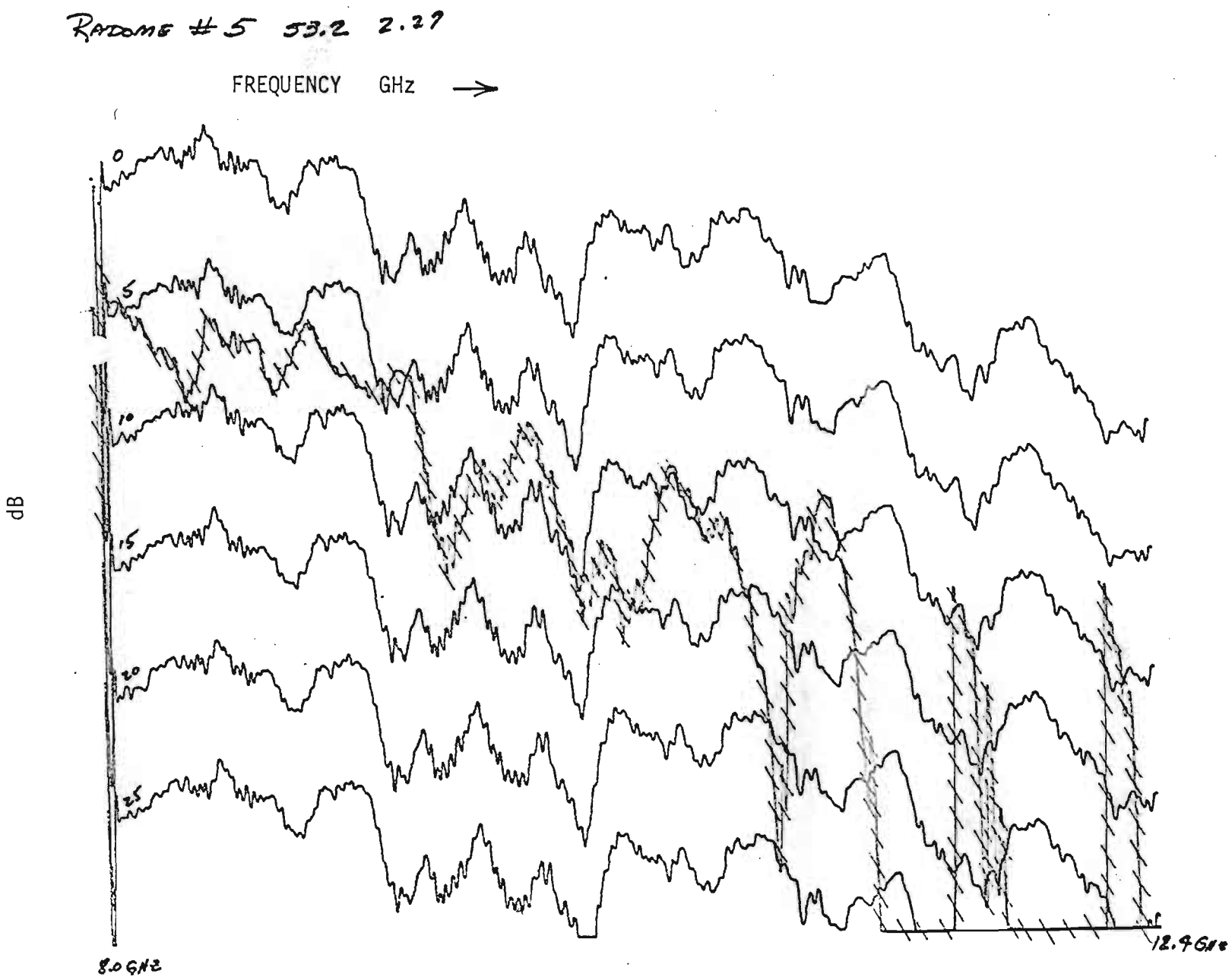

Figure 7. Transmission Through Radome Number 5 (After Exposure to Laboratory Humidity).

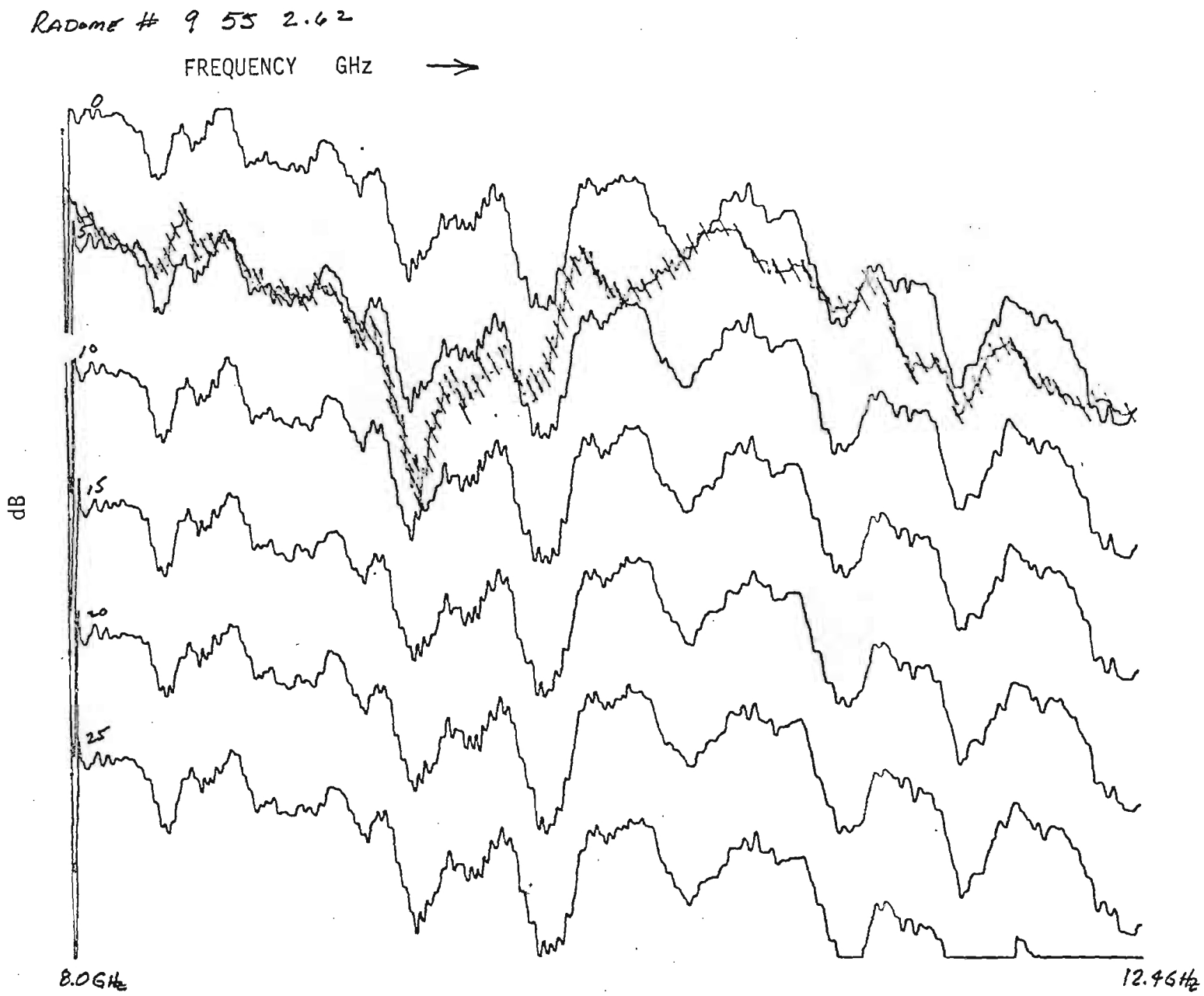

Figure 8. Transmission Through Radome Number 9 (After Thorough Drying).

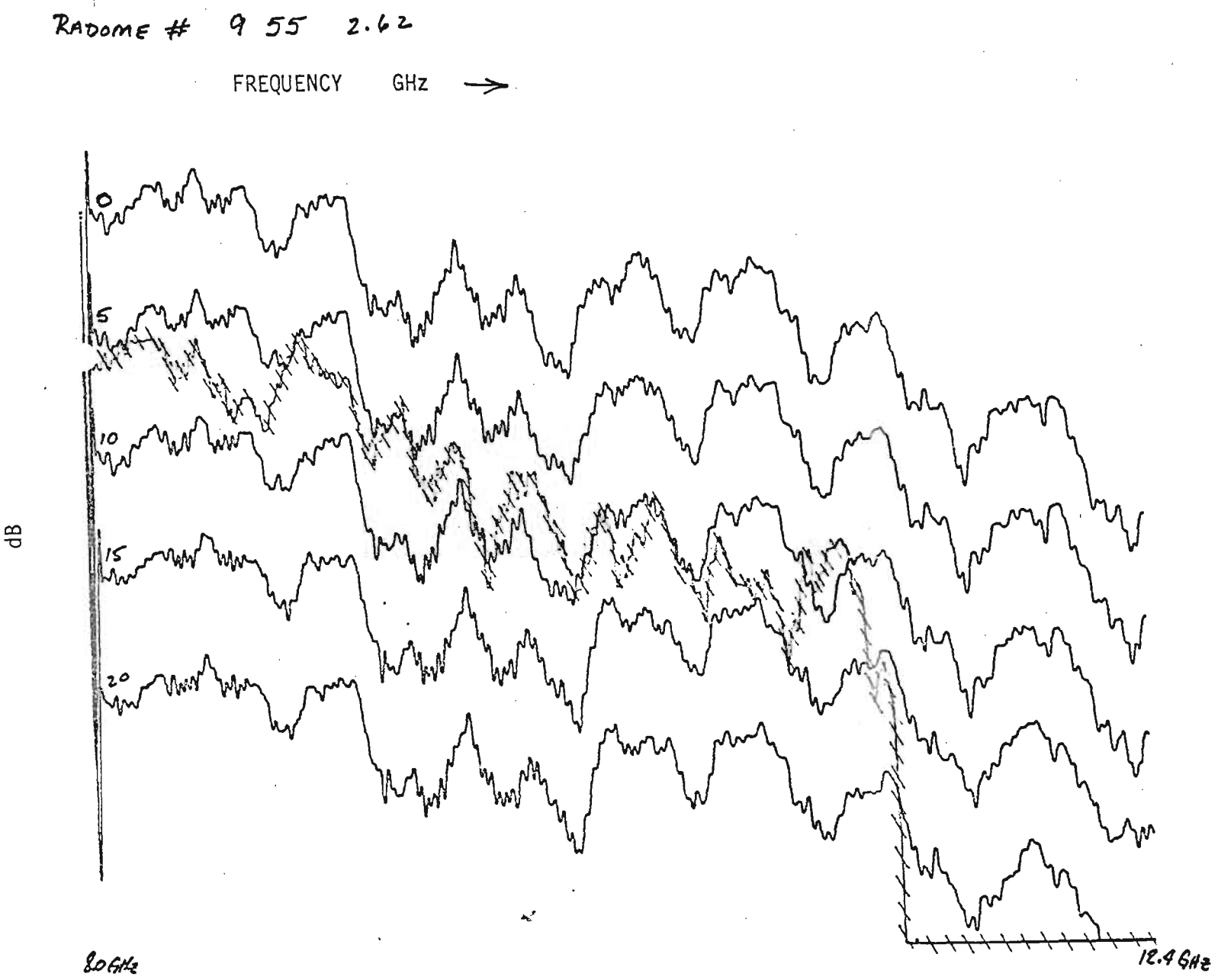

Figure 9. Transmission Through Radome Number 9 (After Exposure to Laboratory Humidity).

AN INVESTIGATION OF REACTION SINTERED SILICON NITRIDE AS A RADOME MATERIAL

FINAL REPORT

(1 March 1975 through 31 August 1975)

By

J.N. Harris

Project A–1724

Prepared Under Contract N00019–75–0372

For

Naval Air Systems Command
(Code AIR–52032A)
Department of the Navy

By

Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332

AN INVESTIGATION OF REACTION SINTERED SILICON NITRIDE AS A RADOME MATERIAL

FINAL REPORT

(1 March 1975 through 31 August 1975)

By

J. N. Harris

Project A-1724

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Department of the Navy

Approved for public release; distribution unlimited.

By

Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332

ABSTRACT

Eleven reaction sintered silicon nitride radomes were prepared by slip casting silicon blanks and reaction sintering in nitrogen to determine the repeatibility of electrical transmission characteristics, examine methods of sealing porous radomes, investigate possible flight attachment systems, and prepare a radome with attachment for rain erosion sled testing. Only two of fifteen castings were lost in the mold. Dry density varied from 1.58 to 1.89 gm/cm^3 due to variation in viscosity and particle size distribution of the slip. Final densities of radomes completing the reaction sintering cycle varied from 2.55 to 2.76 gm/cm^3. Electrical transmission patterns were made on six of the radomes using swept frequency techniques. At the frequency representing a half-wave wall thickness for the particular density of each radome, transmission losses were of the order of a few dB. Weight gained during nitriding was not a good indicator of electrical transmission capability. Acceptable transmission patterns were made on radomes with apparent weight gains as low as 50 percent. Radomes sealed against moisture by impregnating or surface coating with silicone resin were not degraded in electrical performance by the sealing. A dense reaction sintered silicon nitride attachment ring was self-bonded into a lower density reaction sintered silicon nitride radome as a demonstration of the feasibility of this approach for attachment systems. Low expansion carbon composite rings tailored to match the expansion of silicon nitride were also considered for attachment rings. A 36 percent nickel low expansion steel bonded with an epoxy adhesive will be used in an attachment for rain erosion sled testing of a reaction sintered silicon nitride radome at a future date.

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I. PURPOSE

The purpose of Contract No. N00019-75-C-0372 is to continue the research and development efforts of Contract N00019-73-C-0155 and N00019-74-C-0248 to investigate the utility of reaction sintered silicon nitride as a radome material. The objectives are to: (1) Obtain satisfactory electrical properties throughout the radome body, (2) develop a system of sealing to protect the radome from moisture, and (3) develop a simple attachment system to mount a radome on a sled so that rain erosion resistance of an actual radome can be evaluated.

II. INTRODUCTION

This is the third report issued on the development of reaction sintered silicon nitride as a radome material. The previous two reports were issued as the final reports on Contracts N00019-73-C-0155 and N00019-74-C-0248, respectively. The reason for the interest in development of reaction sintered silicon nitride as a radome material is to overcome some of the deficiencies of other ceramic materials which have proven capabilities for high speed flight. Because of the problems associated with rapid heating rates (thermal shock) that accompany the high velocities associated with hypersonic flight only two ceramic materials have proven capabilities for hypersonic radome applications; these are boron nitride and slip-cast fused silica. It is doubtful that boron nitride will be seriously considered for radome applications because of its fabrication limitations and high cost. Slip-cast fused silica is economical to fabricate in large shapes and is capable of providing a radome for surfaced launched systems which can function electrically, thermally and mechanically at low altitudes to velocities above Mach 7, however, its resistance to rain erosion, though superior to boron nitride is less than many other ceramics.

At the present time there is no proven ceramic material capable of providing a radome with adequate thermal shock resistance, good dielectric properties and good rain erosion resistance for air launched systems intended to operate in the Mach 5 to 7 region.

After boron nitride and slip-cast fused silica the material with the best potential thermal shock resistance is reaction sintered silicon nitride. This material is potentially capable of providing a radome with the required

mechanical and electrical properties and should possess substantially better rain erosion resistance than either boron nitride or slip-cast fused silica.

Work on the development of reaction sintered silicon nitride as an engineering material has been conducted mainly in Great Britain since the late 1950's. However, there was only intermittent interest in the potential of reaction sintered silicon nitride as a radome material 1/ because it was generally considered to have inadequate electrical properties 2/.

Reaction sintered silicon nitride was fabricated at Georgia Tech by isostatic pressing and slip casting of commercially available silicon powders. These were then reaction sintered in nitrogen under carefully controlled conditions. Dielectric data on these specimens showed that reaction sintered silicon nitride could be produced with satisfactory dielectric properties for high temperature radome applications.

Having shown that the most important parameter (dielectric properties) for a radome material could be met by reaction sintered silicon nitride, it was felt that the other criteria of importance should be thoroughly investigated to determine whether this material could be satisfactorily used in a radome. The criteria used in selecting a radome material in order of decreasing importance are: (1) dielectric properties, (2) thermal shock resistance, (3) rain erosion resistance, (4) weight, (5) fabricability, and (6) economics. Each of these criteria was examined under Contract N00019-73-C-0155 3/ to determine the suitability of reaction sintered silicon nitride as a radome material. Under that program reaction bonded silicon nitride was fabricated by injection molding, pressing and slip casting from commercial grades of silicon powders containing up to one percent iron and properties were measured on the materials produced. Reaction

sintered silicon nitride was fabricated with densities of 1.6 to 1.65 gm/cm^3 and strength properties were found to be consistent with measurements of other investigators with respect to density 4/. Tests for thermal shock resistance provided thermal stress data consistent with the strength of the material. Impact tests to simulate rain erosion suggested that the slip-cast material should be able to survive rain impact under rocket sled conditions at Mach 5, at a 30 degree angle with only minor damage. Results of dielectric tests indicated that the lower density material was more completely converted to silicon nitride and hence, more consistent dielectric data were obtained with the lower density material. As density increased the difficulty of reproducing dielectric properties between firings also became more difficult.

Under Contract N00019-74-C-0248 5/ improvements were made in the aqueous silicon slips used for slip casting. These improvements eliminated the problem of agglomerates in the slip which proved difficult to nitride in slip-cast parts. These changes in the slip also produced cast parts with much higher green strength than were previously obtained. Under N00019-73-C-0155 all slip-cast parts were formed by drain casting at atmospheric pressure, i.e., after the wall of the cast part reached the proper thickness the remaining slip was drained from the mold and the part allowed to dry. Under Contract N00019-74-C-0248 all radome shapes were match pressure cast. That is, they were cast in plaster molds with an impervious inner mandrel which limited the thickness of the cast. Two sizes of radomes were cast. These were; 5½ inch base diameter by 13 inches high (Mauler) and 8 inches base diameter by 19 inches high (Sparrow). Difficulty occurred in obtaining a high percentage of theoretical weight gain (nitridation)

until late in the program when the source of nitrogen was changed from high pressure (bottled) nitrogen to cryogenic nitrogen. The use of the cryogenic nitrogen resulted in much more consistent nitridation of radomes.

The addition of small quantities (up to 2-3/4 percent) of iron oxide, as pigment grade magnetite, enhanced the nitridation and had no apparent effect on electrical performance. Free space electrical transmission measurements were made on two of the radomes. They showed transmission loss of less than 1 dB and less than 3 dB respectively.

The present program is an extension of the work described above and has as its goals: (1) the attainment of consistent satisfactory electrical properties throughout the radome body; (2) develop a system of sealing to protect the radome from moisture, and (3) to provide a radome complete with simple attachment system capable of being mounted on a sled for determination of rain erosion resistance. The work accomplished on these goals is described in this report.

III. EXPERIMENTAL PROCEDURE

A. Slip-Casting of Radome Shapes

1. Mauler Radome Shapes

The work in progress under contract N00019-74-C-0248 was continued on slip-casting of Mauler radome shapes by pressure match casting. The initial blanks were 0.375 inch in thickness, but part way through the current program 0.100 inch was removed from the master aluminum model used in forming the plaster molds. This resulted in a reduction in wall thickness to 0.275 inch, and is much closer to what a half-wave wall would be in an electrically correct reaction sintered silicon nitride radome in the density range under study. The master model and the precision inner mandrel used to cast the "Mauler" radome shapes were cut using a "hand-made" template for which no equation was available. Therefore, it was not possible to calculate an accurate volume for the cast shape. The estimated dry density was obtained from water absorption and water suspension data on the reaction sintered radomes following the methods of ASTM C-373 for calculating bulk density. Measurements of the radome heights and base diameters before and after nitridation indicated that there was no shrinkage involved. Therefore, the average volume for the 0.375-inch thick wall radome shapes was 920 cubic centimeters and for the 0.275 inch wall shapes was 776 cubic centimeters. These average volumes were used with the dry weights of the radomes to calculate the estimated dry density of radomes 3, 6, 8, 10 and 11. The actual calculated volume from water absorption and suspension data was used to calculate the densities for radomes 2, 5, 7, 9 and 12. On these

radomes the bulk density varied from 2.55 to 2.70 gm/cm^3 and the open porosity from 6.7 to 15.9 percent.

Attempts were made to control the green (dry) density of the silicon casting by adjustments to the viscosity of the silicon slip. Parameters controlling viscosity of the slip are: silicon content, magnetite ($FeO \cdot Fe_2O_3$) content and pH. In some cases viscosity and pH were altered by small additions of monoethanolamine and triethanolamine. Any change in the slip constituents, either silicon or magnetite additions required several days of rolling for the slip to again become stable.

Table I shows data obtained on 15 Mauler castings and the slips used to cast them. As a general rule good castings were always obtained from slips with viscosities of less than 300 centipoise (measured with a No. 2 spindle in a Model LVF Brookfield viscometer at 30 rpm). Some flaw free castings were obtained from slips with viscosities ranging from 300 to greater than 1000 centipoise, but these were the exception rather than the rule. In some instances when slips with viscosities greater than 300 centipoise were used, rapid casting of the slip against the plaster would result in "bridging," leaving a hole in the casting covered by a thin layer of slip. When the radome blank was removed from the mold the "bridged" areas collapsed leaving a dimpled surface with indentions 1/4 to 1/2 inch deep.

Figure 1 is a plot of radome dry density versus viscosity of slip used in casting Mauler radomes of two different wall thicknesses. As a general rule density decreased with increasing viscosity of slip, but as can be seen, there were exceptions to this rule and there was a considerable spread in data.

TABLE I

MAULER RADOMES SLIP-CAST FROM SILICON SLIPS

Radome Number	1	2	3	5	6	7	8	9	10	11	12	15
Date Cast*	4343	5022	5035	5037	5042	5070	5084	5098	5115	5153	5157	5204
Date Slip Made*	4319	5002	5005	5005	5022	5022	5022	5028	5035	5126	5126	5068
Slip Age (days)	24	20	30	32	20	48	66	70	80	27	31	136
Solids Content (w/o)	75	80	75	75	77	75	75	75	75	79.2	79.2	---
Iron Added as Magnetite (w/o)	0	0	0	0	0	2.75	2.75	1.04	0	2.75	2.75	
pH	6.75	6.25	---	6.08	6.1	6.9	7.0	6.45	5.8	6.9	6.9	6.95
Brookfield Viscosity @ 30 rpm (centipoise)	295	379	---	93	280	788	420	82	418	1076	450	300
Dry Weight of Casting (gms)	1516	1491	1612	1610	1593	1446	1265	1357	1469	1252	1311	1446**

(Continued)

TABLE I (Continued)

MAULER RADOMES SLIP-CAST FROM SILICON SLIPS

Radome Number	1	2	3	5	6	7	8	9	10	11	12	15
Dry Density (gm/cm^3)	1.66	1.62	1.75	1.72	1.72	1.58	1.63	1.75	1.89	1.61	1.69	***

NOTES:

1. Radome number 4 was too thin (did not cast fully) no measurements made.

2. Radome number 13 was dimpled and cracked in mold, no measurements made.

3. Radome number 14 broken during attempt to fit argon sintered slip-cast silicon attachment ring into it. Final dry weight before nitriding not measured.

* Julian dates.

** 1446 represents weight of "machined" unfired radome and argon sintered attachment ring combined.

*** Not possible to estimate dry density with modifications to radome.

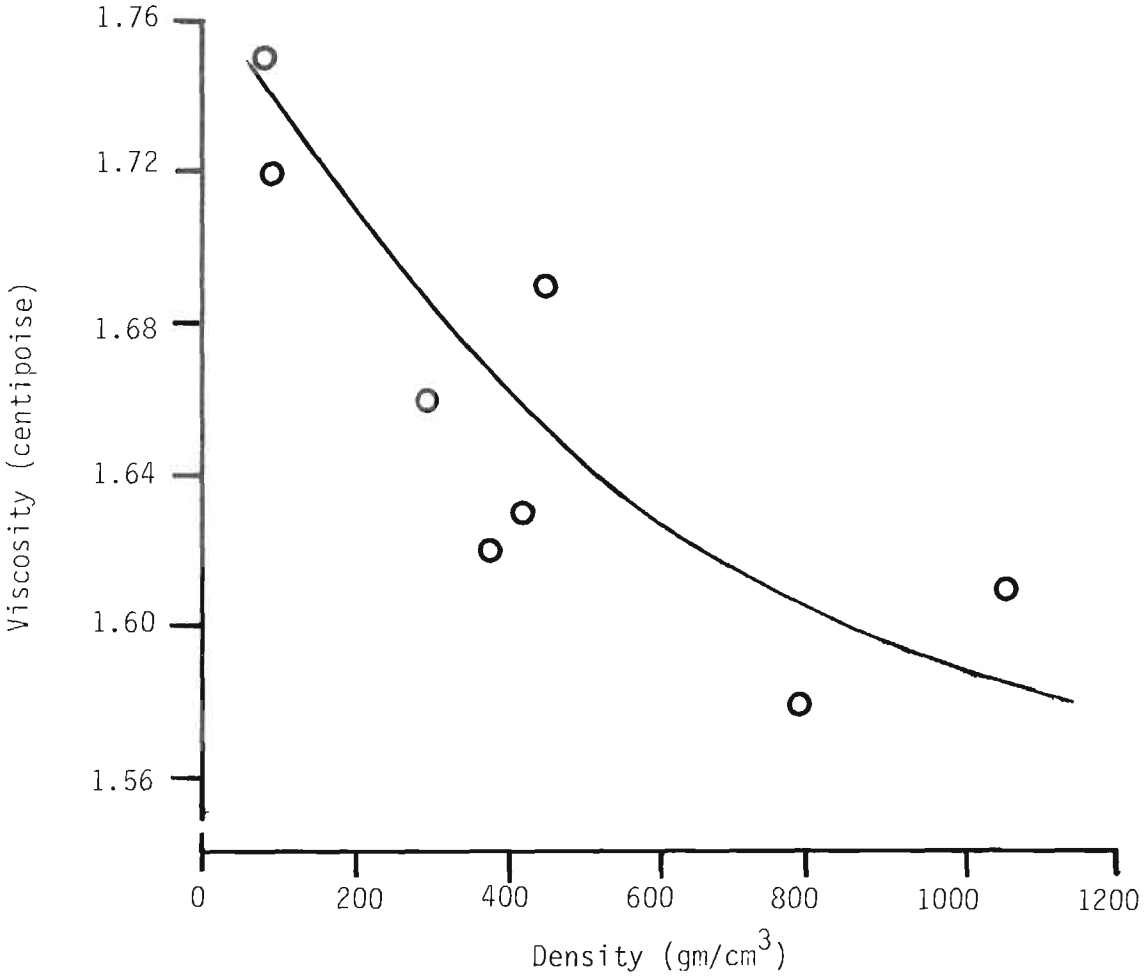


Figure 1. Dry Density of Radome Casting versus Viscosity of Slip Used.

2. Sparrow Radome Shapes

Sparrow radome blanks were cast in tooling previously used for slip-cast fused silica radomes which produced blanks with a 0.49 inch wall and in modified tooling which produced a 0.27 inch wall. No problems were encountered in casting blanks (with < 300 centipoise viscosity slips) and these blanks had sufficient green strength for ease of handling. Unfortunately, it was not possible to reaction sinter any of the Sparrow radomes due to problems with the retorts.

B. Reaction Sintering of Radome Shapes

1. Mauler Radomes

Late in the previous contract period 5/ it was discovered that more consistent nitriding could be obtained using liquid nitrogen as the source of nitrogen rather than bottled gas. Liquid nitrogen was used for all nitridation under this contract. Although weight pick-up still varied, results were much more consistent than on the previous contract.

The furnace and associated equipment are shown schematically in Figure 2 and photographically in Figure 3. This is the same set-up as used in the previous program with the exception that a programmable temperature controller was added. This resulted in more flexibility in time and temperature control and more consistency from firing to firing.

Several reaction sintering temperature-time cycles were investigated, but the most complete conversion to silicon nitride was obtained using the following four step schedule on radome blanks containing 2-3/4 percent magnetite ($FeO \cdot Fe_2O_3$).

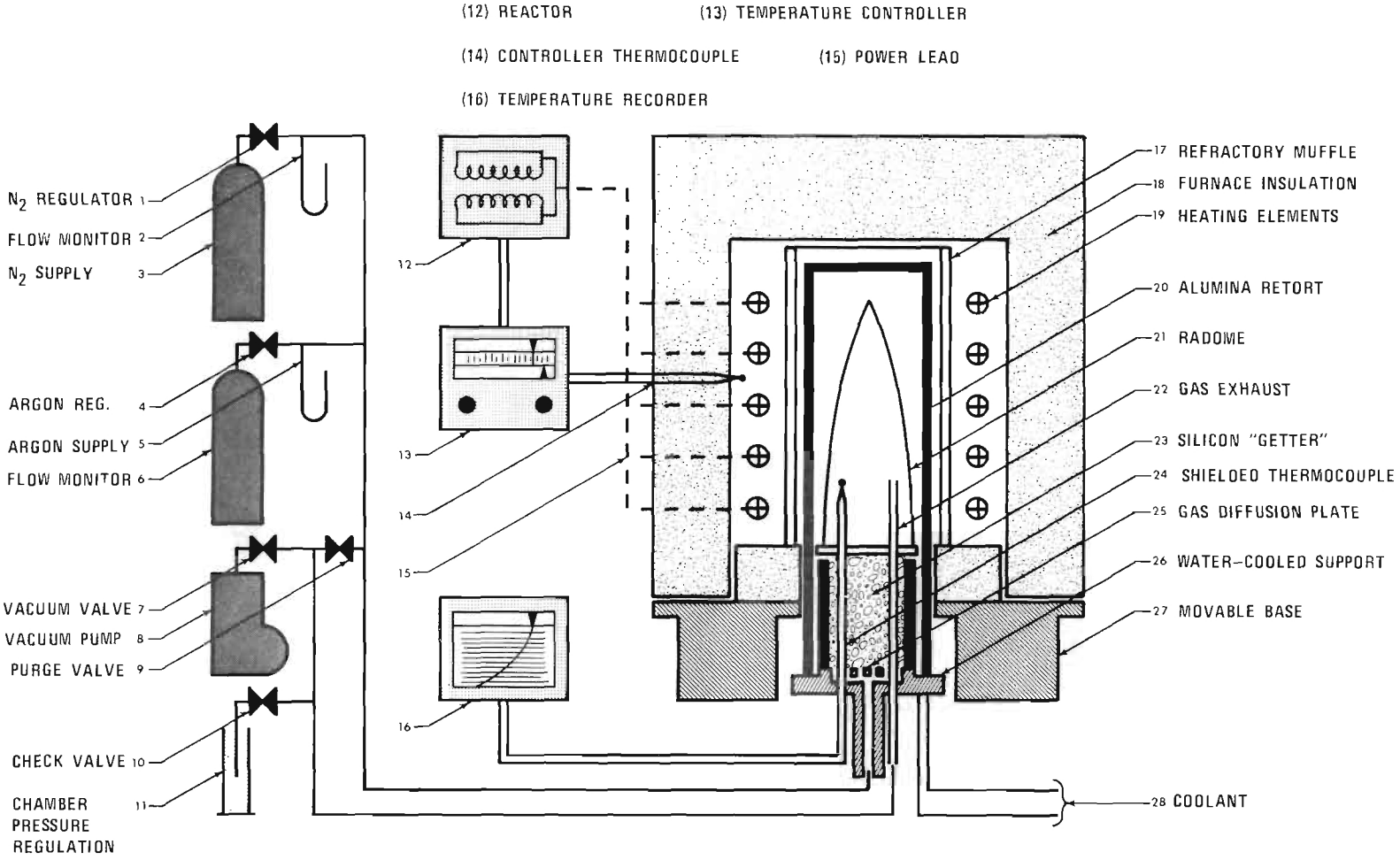
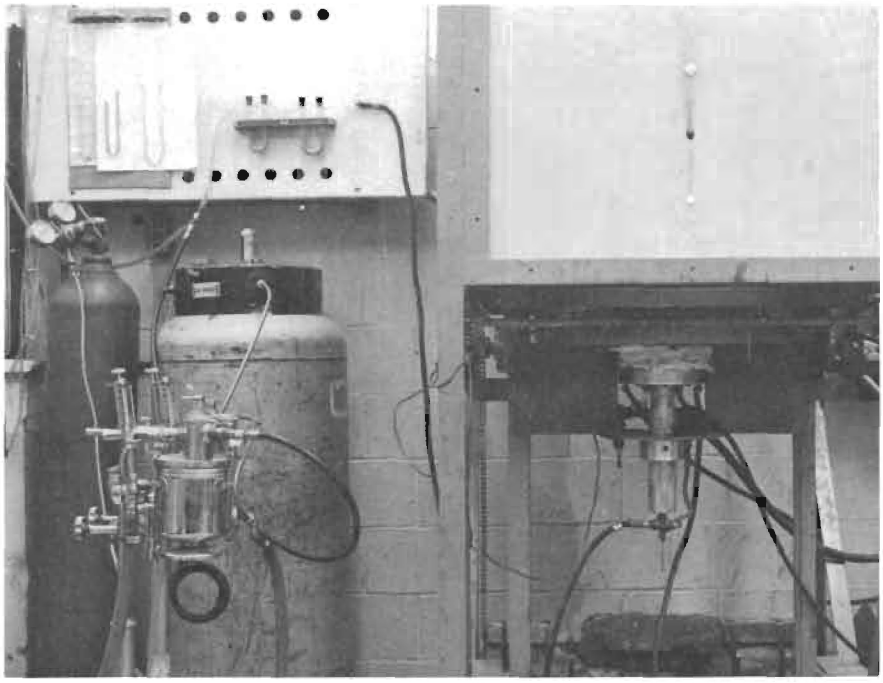
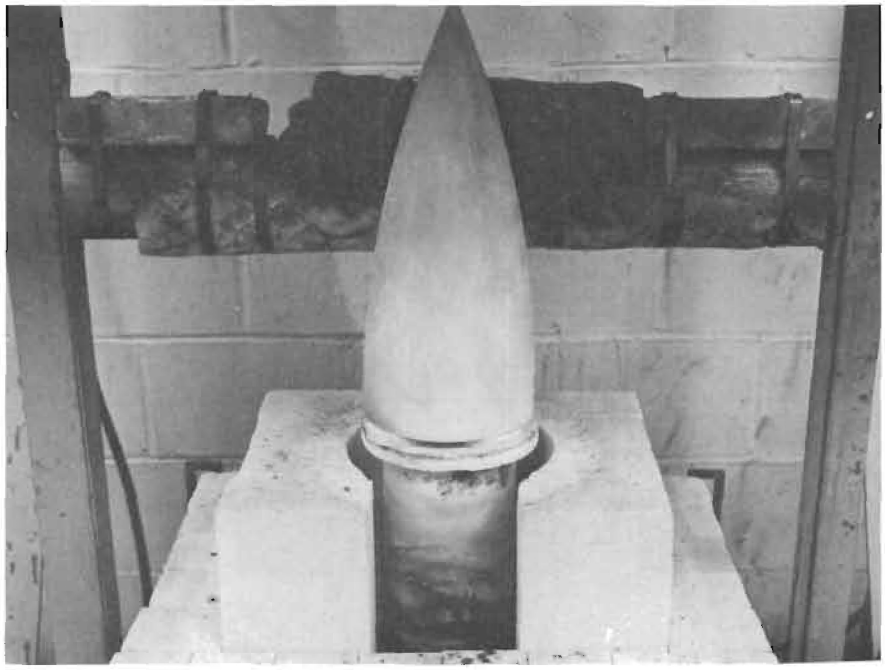


Figure 2. Reaction Sintering Facility



(a) Reaction Sintering Furnace and Associated Equipment



(b) Mauler Radome with Retort and Muffle Removed.

Figure 3. Reaction Sintering Facility Showing Furnace in Operating Position and Mauler Radome on Firing Pedestal.

Room Temperature to 2000^{o} F	16 hrs
Hold at 2000^{o} F	2 hrs
2000^{o} F to 2300^{o} F	2 hrs
Hold at 2300^{o} F	24 hrs
2300^{o} F to 2400^{o} F	2 hrs
Hold at 2400^{o} F	6 hrs
2400^{o} F to 2550^{o} F	2 hrs
Hold at 2550^{o} F	24 hrs
Cool Slowly to Room Temperature	

Temperatures indicated above are those recorded by the shielded thermocouple located inside an alumina tube inside the retort. Actual temperature of the radome was slightly higher than the indicated temperature.

Sintering data obtained for Mauler radomes are shown in Table II. Differences in weight gain and final bulk density after nitridation are attributed to differences in cast density, iron content and, in some cases, interrupted sintering cycles due to equipment malfunctions (usually leaks or stoppages in the nitrogen flow).

C. Sealing of Reaction Sintered Silicon Nitride Radomes

Due to the porous nature of reaction sintered silicon nitride radomes they must be sealed to prevent moisture pick-up and a subsequent degradation of electrical transmission. Several methods of sealing were considered, however, due to the limited number of electrically good radomes the number of actual systems tested had to be limited.

TABLE II

MAULER CASTINGS REACTION SINTERED

Radome Number	2	3	5	6***	7	8	9	10	11**	12	15*
Dry Weight of Casting (gms)	1491	1612	1610	1593	1446	1265	1357	1469	1252	1311	1446
Dry Density (gm/cm^3)	1.62	1.75	1.72	1.72	1.58	1.63	1.75	1.89	1.61	1.69	
Nitrided Weight (gms)	2422	2417	2468	2134	2327	2036	2103	2147	1859	2088	2260
Percent Weight Gain	62.4	49.9	53.2	38	60.9	60.9	55	46.1	48.5	59.3	56.2
Nitrided Density (gm/cm^3)	2.63	2.63	2.63	2.30	2.55	2.62	2.71	2.76	2.40	2.69	

*Includes bonded silicon nitride attachment ring. Estimate of green and nitrided composite density not made.

**Reaction sintered to 2350^{o} F only. ***Extensive Boil Out of Silicon.

1. Inorganic Coatings

One method considered was to seal the radome by chemical vapor deposition (CVD) of silicon nitride, however, any such coating would have to remain crack-free to prevent moisture penetration. Unfortunately, equipment was not available to accomplish this task in-house and a suitable sub-contractor was not found.

A second method considered was the impregnation of the reaction sintered silicon nitride radome with colloidal silica followed by the subsequent fusion of the silica into a glassy coating at a temperature above the melting point of the silica and below the decomposition temperature of the silicon nitride. A limited attempt was made to use this technique by impregnating the forward section of a reaction sintered silicon nitride Mauler radome shape with colloidal silica. A total of 16 grams of silica was picked up. The radome was thoroughly dried, then sintered in a natural gas fired kiln to a temperature of 3300^{o} F. It was then allowed to cool in the kiln to room temperature. The sintered radome had a very glassy appearance; however, upon soaking the treated portion in water there was a weight pick-up of more than 100 grams indicating that the radome was not sealed.

2. Impregnation Treatments

Another approach to sealing of reaction sintered silicon nitride radomes is one which has been used successfully with slip-cast fused silica radomes. In this technique the porous radome structure is impregnated with a silicone resin and is cured. The resin seals the structure effectively at low temperatures and will burn out at high temperatures. Since a missile

radome is a one time use item, the resin is no longer needed after it reaches the high temperatures created by high velocity flight. Since the resin has a low dielectric constant, it should not affect deleteriously electrical transmission properties. Reaction sintered silicon nitride radomes with good electrical transmission characteristics were impregnated with General Electric SR-80 resin. The procedure for impregnation was to thoroughly dry the radome at 350° F to remove all moisture. The radome was then placed tip down in a metal container. The area outside the radome was filled with General Electric SR-80 silicone resin. The open end of the radome was covered to maintain a high resin solvent vapor pressure on the inside of the radome. This is an important step otherwise the solvent evaporates as it is soaking through the radome wall and the residual solids block the pores preventing complete impregnation. A soak period of 72 hours was used to assure penetration of the resin. In the case of slip-cast fused silica complete impregnation can be determined because the entire radome wall becomes translucent. This is not the case with reaction sintered silicon nitride. Complete penetration can only be determined by a build-up of "dampness" on the inner wall. After impregnation the radome is heated to 250° F for one hour to evaporate the excess solvent. The radome is then heated to 480° F for two hours to cure the resin.

Radome number 7 had an open porosity of 15.9 percent and picked up 60 grams of weight after impregnation. The resin impregnant was 2.5 percent of the total radome weight.

The cured radomes were carefully weighed, soaked in water and reweighed. No measurable weight gain was noted indicating the radomes were completely sealed.

3. Surface Sealing Coatings

An alternate method of using silicone resin as a sealant is to use it as a coating on the inside and outside of the radome. The advantage of this sealing system is that there is less penetration into the radome and hence, less material to form a char at elevated temperature. A potential disadvantage is that it is more likely to be scratched in service, providing a potential entry point for moisture.

Dow Corning 808 resin was used to seal a Mauler radome. The 808 resin was poured into the inside of the radome and then poured out leaving a continuous film on the inner surface. The outer surface was coated by brushing. The resin coating on the radome was cured by heating to 250^{o} F for one hour to drive off the excess solvent. It was then heated to 390^{o} F for two hours to complete curing. Weight gain was only 10 grams or less than one-half percent of the total weight. This cured radome was also subjected to a water-soak to determine if any moisture would be picked up. No weight gain occurred after soaking.

D. Electrical Transmission Measurements

A free-space transmission measurement was used to determine transmission normal to the radome wall. Each radome was mounted in an anechoic chamber whereby unwanted reflections from surrounding objects were at a minimum. A broadband antenna was placed inside the radome perpendicular to the radome axis; thus, the antenna was "looking" through the sidewalls and not through the nose. A pick-up antenna was located in the far field of the radome antenna. Using swept-frequency techniques, transmission data on the radomes were obtained from 8 to 12 GHz. No attempt was made to correct

for reflections within the free-space transmission system, since it was felt that the measurements as made would be adequate for comparison purposes.

Five unsealed radomes were tested for transmission capability. Three radomes (numbers 3, 5 and 12) were measured immediately after thorough drying and then again after sitting in the laboratory for several days ($\sim 75^{o}$ F and 60 percent relative humidity). Transmission was somewhat attenuated by moisture absorption, however, a direct comparison is difficult because the antenna may not have been located in the same position with respect to the radome for the two test conditions. Transmission patterns for all radomes and test conditions are shown in the Appendix.

At the particular frequency where the radome was near a half wavelength in thickness, two of the silicone resin sealed radomes had good transmission properties before and after sealing with resin. This occurred near 9.3 GHz as noted in Figure 15 in the Appendix. One of the radomes sealed with GE SR-80 impregnating resin could not be measured, since no transmission pattern could be attained after sealing. Either the radome numbers were mixed up before sealing and an electrically bad radome was sealed or a good radome was not thoroughly dried before impregnation.

E. Attachment Systems

1. Flight Attachments

The development of a flight worthy attachment system for a reaction sintered silicon nitride radome would represent a complete research program. A short effort was devoted, however, to one possible concept, the use of a dense reaction sintered silicon nitirde attachment ring bonded into a less dense reaction sintered silicon nitride radome.

A threaded attachment ring with an internal 8-pitch thread was slip-cast from a silicon slip designed to produce a dense casting ($\sim$ 1.75 gm/cm^3 green density). This ring was cast in a plaster mold with an aluminum threaded insert placed in the center of the mold as shown in Figure 4. Figure 5 is an exploded view showing the Benelex master pattern used to form the plaster mold, the plaster mold, the multi-piece aluminum insert, and a silicon ring cast in this mold. The casting procedure was to assemble the mold as shown in Figure 4, fill the annulus with silicon slip and to keep it filled until fully cast. When cast, but before drying, the aluminum plunger was removed from the center of the threaded aluminum section and the segmented threaded sections moved inward to release them from the casting. This section was then removed from the mold. The cast piece was also removed from the mold, dried and sintered under argon to 2000^{o} F for 6 hours. This sintered ring was then fitted to a dry slip-cast silicon Mauler radome shape. Imperfections and slight out-of-roundness were corrected by alternate light sanding on the dry radome shape and on the argon sintered silicon ring. Before final fitting the outside of the ring and the inside base of the radomes were painted with silicon slip. The attachment was placed in the base of the radome. The combination was reaction sintered following the schedule given in Section B. After sintering the attachment was firmly bonded to the radome. There was no visual evidence of a line separating attachment and radome. The finished radome is shown in Figure 6.

2. Rain Erosion Sled Attachments

The original intent of this portion of the program was to prepare a Sparrow size radome with an attachment suitable for rain erosion sled

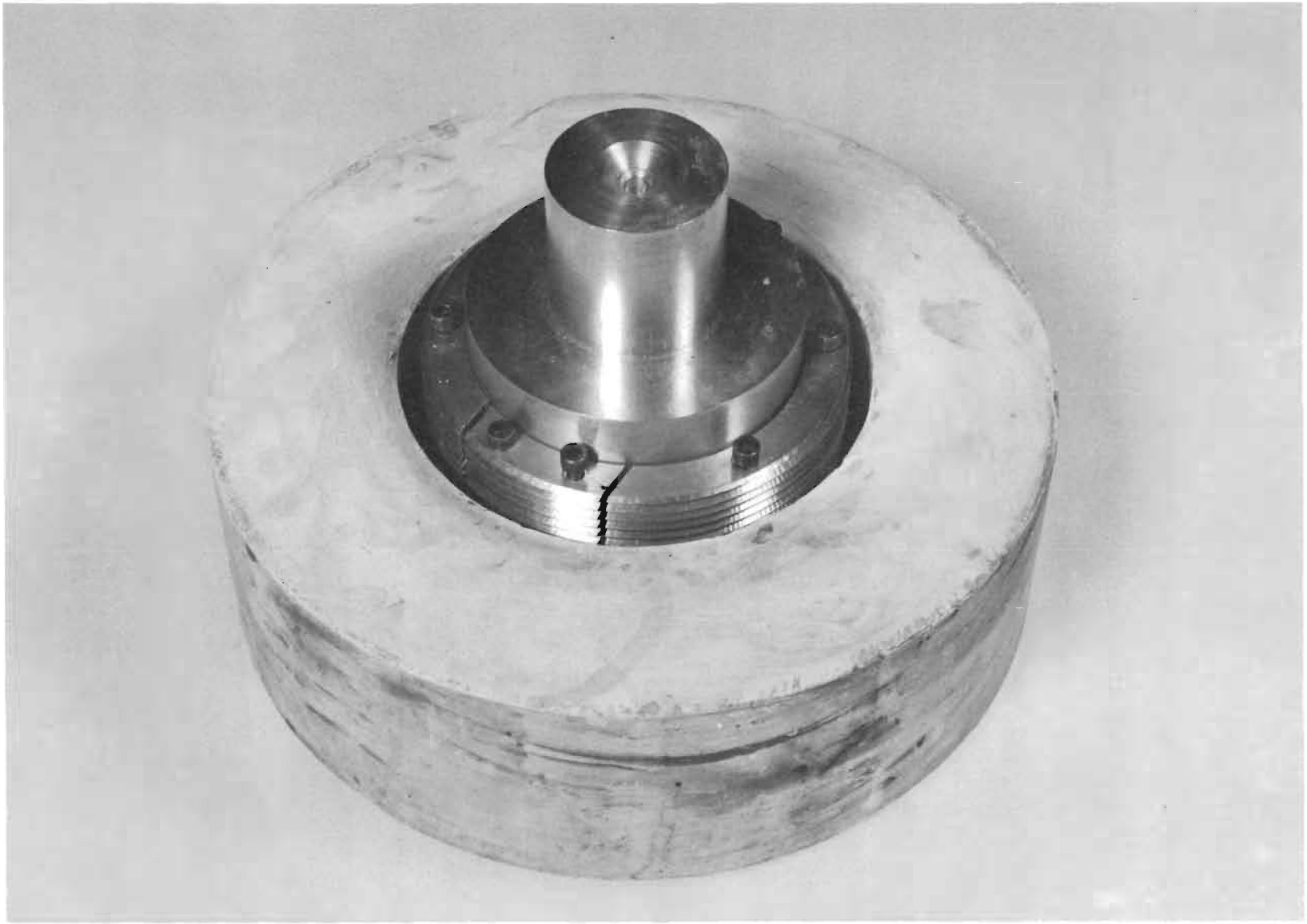


Figure 4. Assembled Mold for Slip Casting Threaded Silicon Attachment Ring.

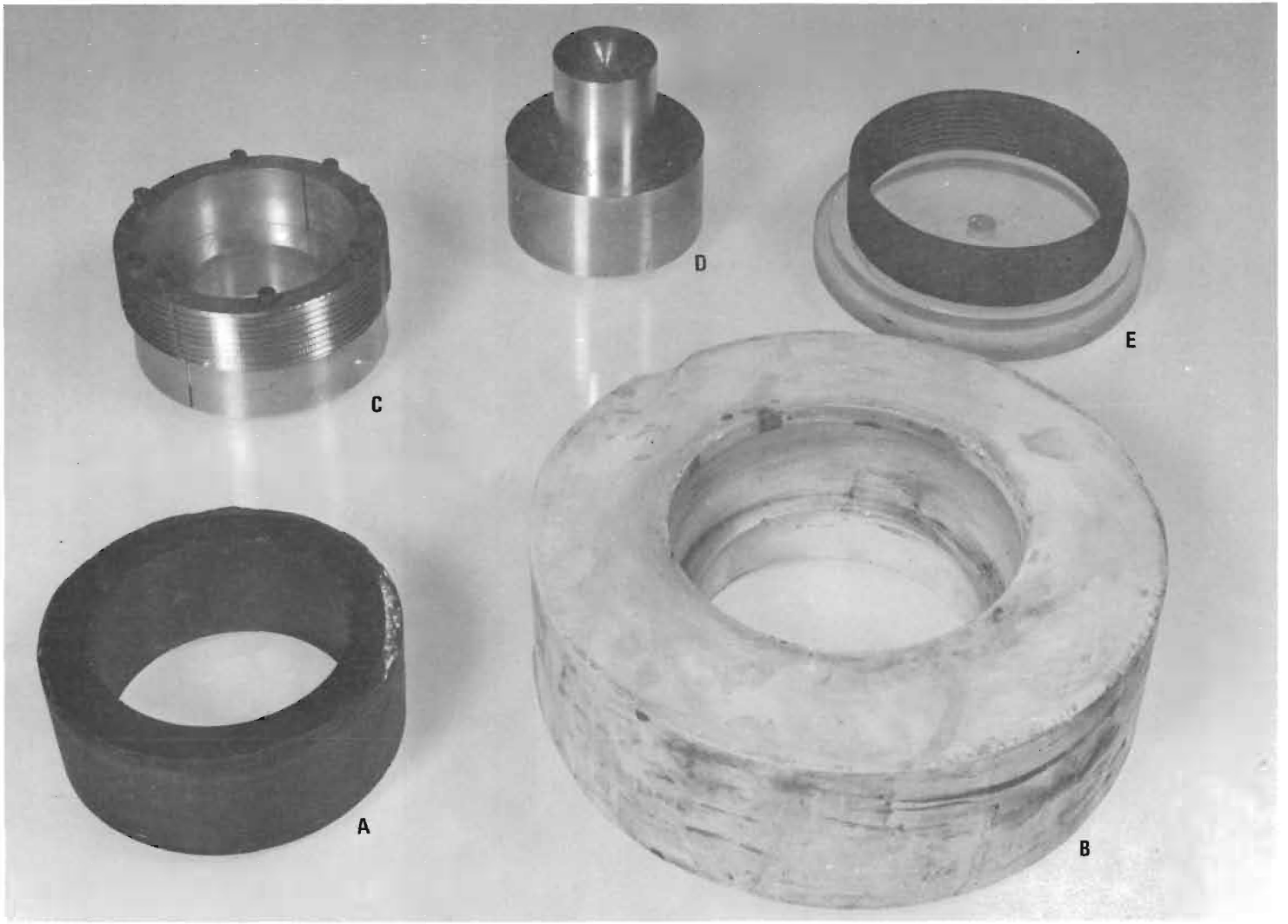


Figure 5. Disassembled Mold Showing Parts Required and Slip-Cast Silicon Ring. (A) Master Model for Plaster Mold, (B) Plaster Mold, (C) Segmented Threaded Ring, (D) Aluminum Pattern for Adjustment of Segmented Ring, (E) Plastic Mold Base and Slip-Cast Silicon Ring.

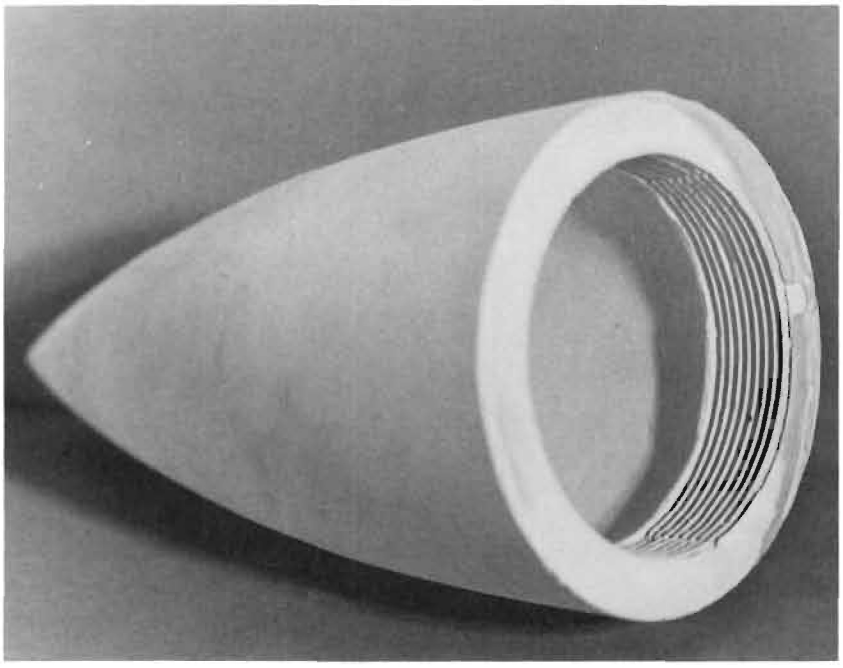


Figure 6. Reaction Sintered Silicon Nitride (RSSN) Radome and RSSN Attachment Ring.

testing at Mach 4. The intent was to provide a radome to the Air Force Materials Laboratory suitable for attachment to one of their sleds. The Air Force would have tested this radome at some future date. The difficulties occurring with the retort on the large firing facility precluded the preparation of a Sparrow size radome. An Invar® forging was on hand and a design of an attachment previously used in slip-cast fused silica Sparrow radomes would have been used. The design of this attachment is shown in Figure 7.

When it became evident that a Sparrow size radome could not be reaction sintered in the existing facility, an Invar forging small enough to fit a Mauler size radome was ordered. This forging did not arrive before the end of the contract. Communication 6/ was made with the Air Force Materials Laboratory concerning design of the Mauler attachment for attachment to an Air Force sled. The result of this communication was that means of attachment to the sled was not important, since some type of transition piece would be required to mount the smaller Mauler radome. Also, the Mauler radome could not be tested unless there was some requirement for a small pay-load on a sled. The attachment placed in the Mauler radome will follow one of the designs of Figure 7.

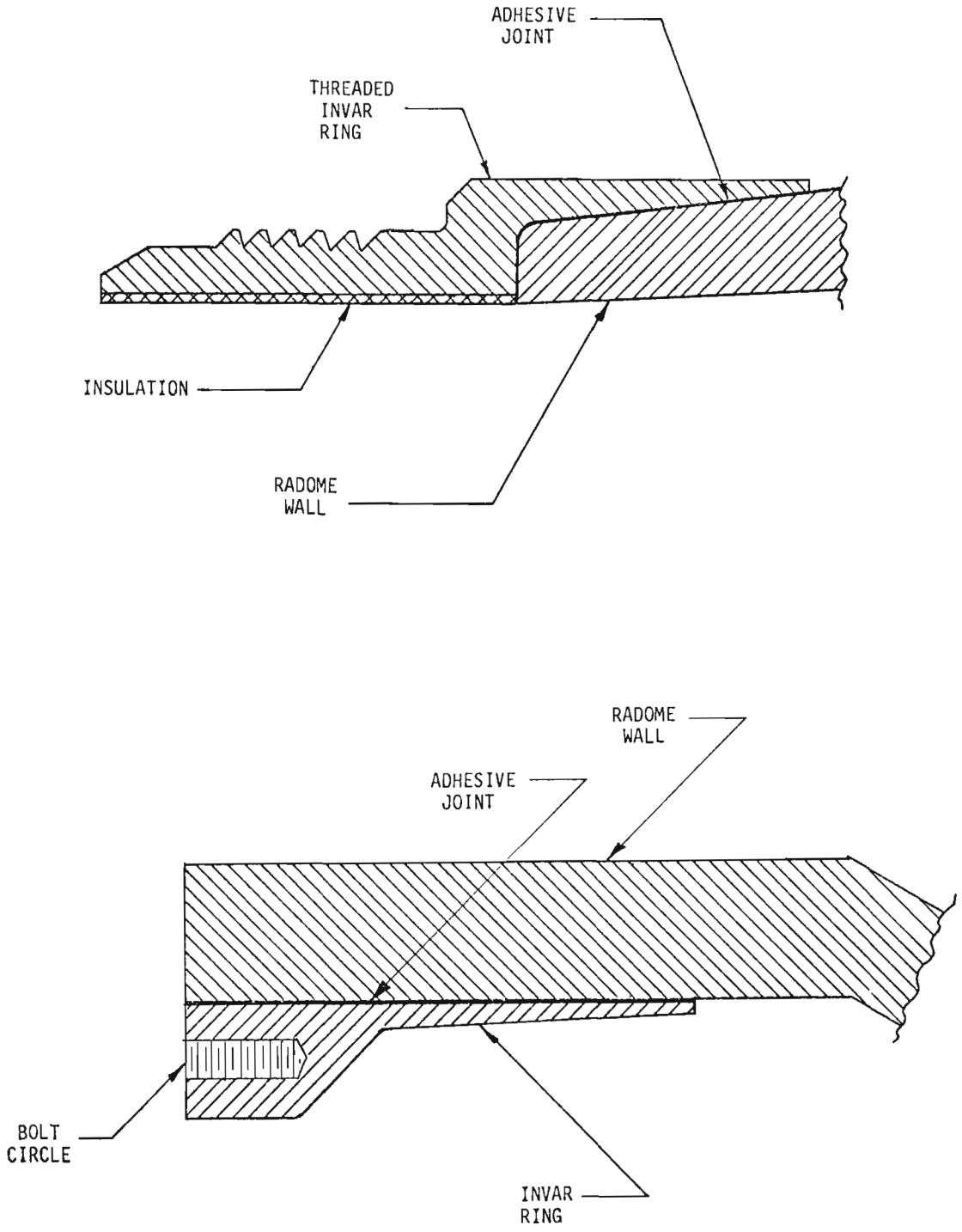


Figure 7. Typical Attachment Designs for Radome Rain Erosion Sled Testing.

IV. DISCUSSION

A. Slip-Casting of Radome Shapes

Based on the estimated dry densities obtained the precision pressure casting technique produced a higher density casting than non-pressure casting of a flat plate from the same slip at the same time. This is the reverse of the situation with pressure slip-cast fused silica and the difference is apparently greater with silicon castings. The pressure cast radomes are denser than non-pressure cast flat plates by 0.05 to 0.10 gm/cm^3. As a result of this difference all electrically acceptable slip-cast reaction sintered silicon nitride radomes had a final density of the order of 2.6 gm/cm^3 rather than a density in the range of 2.3 to 2.5 gm/cm^3 which had been sought for ease of nitridation and potential survival of rain at Mach 4.

The factors influencing the cast density of the precision pressure cast radome shapes are the plaster mold and the general mobility of the particles or viscosity. The viscosity is controlled by: particle size and distribution, solids content of the suspension, chemical additives and pH. For the radomes shown in Table I viscosity was controlled by increasing or decreasing solids content, changing the particle size distribution by the addition of pigment grade magnetite and changing the pH by additions of monoethanolamine and triethanolamine. Figure 1 is a plot of viscosity versus dry density for the radomes listed in Table I. As a general rule it appears that some control of density can be achieved by changing the viscosity, but as can be seen a considerable spread in data was obtained for radomes cast with slips of essentially the same viscosity. For example,

radomes 2, 8, 10 and 12 had viscosities between 379 and 450 centipoise. However, dry density of these radomes varied from 1.62 to 1.89 gm/cm^3. Parameters other than viscosity must also be considered. For example, of this group radome 2 had the lowest cast density (1.62 gm/cm^3). It was made from a slip with the highest solids content and no magnetite. Radome 10 was made from a similar slip except it had a lower solids content and it had the highest dry density of the group. Although the two slips were similar except for solids content they were prepared from silicon material dry ground at different times. The two slips showed a significant difference in pH and there could have been a significant difference in particle size distribution. The other two slips in this group (radomes 8 and 12) were similar in pH and both contained 1.75 percent Magnetite. However, the slip for radome 8 contained a small amount of monoethanolamine and had 4.2 weight percent less solids than the slip for radome 12. Radome 8 had a slightly lower density than radome 12 (1.63 versus 1.69 gm/cm^3) which is the reverse of what would have been expected.

Radomes 6, 7 and 8 were all cast with the same basic slip. Radome 6 contained no magnetite. The slip used to cast it had a relatively low viscosity 280 centipoise and the radome, as expected, had the highest cast density. This slip was modified by adding 2.75 weight percent magnetite and cutting the total solids from 77 to 75 by adding water. This resulted in a slip with a high viscosity (788 centipoise) and produced a dry radome with the lowest density obtained. However, the slip was too viscous and "bridging" of slip caused by fast casting resulted in voids behind the plaster wall which resulted in a "pock" marked radome. Small additions of monoethanolamine

to this slip over a 14 day period resulted in decreasing the viscosity to 420 centipoise. This caused an increase in density with the casting of radome 8 to 1.63 gm/cm^3. This radome also had "pock marks."

At least one radome (number 12) was cast with a very high viscosity slip and was essentially a perfect casting with the second lowest density obtained. Although the viscosity was high (1076 centipoise) as measured with the Brookfield viscometer, the rheology of this slip was different from previous high viscosity slips in that it had a "creamy feel" and flowed readily. Four days later radome 13 was cast with the same slip, but at this time the viscosity had decreased to 450 centipoise. This change resulted in a dry density increase from 1.61 to 1.69 gm/cm^3. One major difference between the slip used to cast radomes 12 and 13 and the slip used to cast radomes 7 and 8 was the time at which the magnetite was added. In the case of the slip used for radomes 7 and 8 the magnetite was added sometime after the initial slip had been made for more than 20 days. A considerable amount of water had to be added with the magnetite to bring the slip down to a working consistency which resulted in a drop in total solids content from 77 to 75 percent. In the case of the slip used for radomes 11 and 12 the magnetite was added from the beginning. That is, a portion of the silicon and magnetite were added to the water, rolled until thoroughly dispersed and another portion of silicon and magnetite added. This procedure was repeated until a workable slip was achieved containing 79.1 percent solids. This resulted in a high solids, high viscosity slip, but with a rheology completely different from the rheology obtained when magnetite was added to an existing slip.

One additional uncontrolled parameter was that radomes 1 through 10 were made from slips from one batch of dry ground silicon powder. Radomes after number 10 were made from slip from a different batch of powder. The same grinding procedures were used, but due to grinding, and a different time of year (laboratory humidity-temperature differences) there may have been some slight differences in particle size distribution which produced slips with higher solids content and apparently different rheologies.

The question of whether slip age at time of casting had any effect on properties of the dry casting or on the ability to completely reaction sinter the casting could not be answered due to the other variables described above. If future studies are conducted these areas should be covered.

B. Reaction Sintering of Radome Shapes

No major problems were encountered in reaction sintering of Mauler size slip-cast fused silica radomes using the firing schedule shown under III Experimental Procedure. The differences in percent weight gained and final nitrided densities are attributed to differences in the silicon castings, with the exception of radomes 6 and 11. Radome 11 was only reaction sintered to 2350^{o} F due to a leak in the nitrogen system. Radome 6 had extensive boil-out of silicon due to an intentional attempt to perpetuate an exothermic reaction by supplying all the nitrogen required once the reaction was started. All other exothermic reactions were "quenched" by holding the nitrogen flow constant and maintaining 7-1/2 inches water pressure on the system by adding argon.

Numerous investigators have reported the on-set of an exothermic reaction at various temperatures, however, in these radome firings only one exotherm was noted and it always occurred at the same temperature as recorded by the alumina shielded platinum-platinum rhodium thermocouple inside the retort. The reaction always began at 2060^{o} F as evidenced by a sudden drop in chamber pressure. If chamber pressure was restored at this point by the introduction of argon no evidence of a change in rate of temperature increase was recorded by the alumina shielded thermocouple and argon flow could be shut off when the temperature reached 2300^{o} F. However, nitrogen flow had to be increased slightly to maintain the pressure head. No changes were necessary in the nitrogen flow when the temperature was increased to 2400^{o} F or to 2550^{o} F. In the case of radome number 6 when pressure began dropping in the chamber at 2060^{o} F the flow of nitrogen was increased significantly and temperature recorded by the shielded thermocouple rose quickly to 2300^{o} F. At this point nitrogen flow was decreased and argon added to maintain the retort pressure. The retort temperature dropped until such time as the increased furnace temperature brought the temperature back to 2300^{o} F. At this point the argon was shut off and the firing continued normally. At the end of this firing extensive boil out was found as evidenced by more than 60 grams of melted silicon in the bottom of the retort. This indicates that the actual radome temperature exceeded 2600^{o} F during the time the slow responding enclosed retort thermocouple reached an indicated temperature of 2300^{o} F.

A theoretically completely reaction sintered silicon nitride radome would show a percent weight gain of 66 percent. However, in actuality some silicon is vaporized and plated out in the retort and a weight gain of

62 to 63 percent is usually considered complete conversion. Only one radome, number 2, reached this level with an indicated 62.4 percent weight gain. Two other radomes approached this weight gain, numbers 7 and 8, with a weight pick-up of 60.9 percent. One radome, number 12, had a weight increase of 59.3. All of these radomes had transmission patterns with low losses in the part of the E-M spectrum examined.

Radomes 9 and 10 had poor weight pick-ups and final densities of greater than 2.7 gm/cm^3. The firing cycle used was not sufficiently long for radomes of this density.

Radomes 3 and 5 had low apparent weight gains, but final densities of 2.63 gm/cm^3. The electrical transmission pattern for radome 3 was as good as the pattern for radome 2 and the pattern for radome 5 was only slightly poorer. The good transmission patterns are not understood for these radomes unless an error was made in the original dry weights of these two radomes.

It is not anticipated that any problems would have occurred in reaction sintering the Sparrow size radomes, since the facility intended for use was essentially a scale-up of the one used for the Mauler radomes. The problems of thermal shock with the alumina retorts and the breaking of the one silica retort prevented any attempt at reaction sintering of Sparrow shapes.

C. Sealing of Reaction Sintered Silicon Nitride Radomes

The purpose in sealing porous, reaction sintered silicon nitride radomes is to prevent moisture penetration and the subsequent degradation of electrical characteristics. The ideal coating would be a CVD layer of silicon nitride, but, since it was not possible to investigate such a coating

under this contract other concepts were investigated. The possibility of fusing deposited colloidal silica in the pores of the silicon nitride to provide a glassy coating was investigated by heating the impregnated radome above the melting point of silica. After cooling the radome appeared to have a vitreous surface, but water absorption indicated that the surface was either microcracked or there was insufficient glassy silica to seal off the pores. Two silicone resin systems were investigated which have been used successfully in sealing slip-cast fused silica radomes without degrading electrical transmission. Both of these systems have advantages and disadvantages. The first is Dow Corning 808 silicone resin. This is a surface sealant that penetrates only a short distance below the surface. Since it is a surface coating it will burnoff the surface during high velocity flight and any char will be oxidized. Thus, there will be nothing left to degrade transmission. Disadvantages are all exposed surfaces, inside and out must be coated to assure no moisture penetration. There is also the remote possibility of a surface scratch deep enough to penetrate the coating, thus providing a path for moisture penetration.

The second resin system, General Electric SR-80 silicone resin, is an impregnating type system which penetrates throughout the porous structure. An advantage is that it would be very difficult to damage to the point of moisture penetration. A disadvantage is that, depending upon the flight conditions, sufficient char may form in the porous structure to degrade electrical transmission.

D. Electrical Transmission

The small attenuation of the transmitted signal on several of the radomes tested shows the feasibility of fabricating electrically acceptable

reaction sintered silicon nitride radomes by slip casting of silicon metal powders and subsequent reaction sintering. A comparison of figures in the appendix will show the differences in transmission for thoroughly dry radomes and for those containing some trace of moisture. The detrimental effects of moisture on transmission can be seen. Figure 15 shows the electrical transmission of two radomes sealed with two different silicone resins. Both show good transmission properties at the particular frequency where the radome walls were near a half wavelength in thickness. These two radomes had equally good transmission properties before and after treating. The best transmission occurred near 9.3 GHz as noted in Figure 15 No attempt was made to correct for reflections within the free-space transmission measurement system, since it was felt that the measurements as made would be adequate for comparison purposes. Because of this the expected resonant frequency shift caused by impregnating the wall was not noted. This shift would be very small and can be calculated by knowing the dielectric constant of the material before and after impregnating with the silicone resin.

Insufficient time and funds were available to investigate the effect of chars formed on heating on the electrical transmission characteristics. Whether char will be formed or the amount of char formed will depend on the flight conditions of a particular missile. These characteristics should be known and heating rates occurring in flight should be used to properly evaluate whether or not char formed is detrimental to electrical transmission of a particular radome.

E. Attachment System

1. Flight Attachment

The demonstration reaction sintered attachment ring bonded into the reaction sintered radome shows the feasibility of this concept. This concept could also be used to bond a hot pressed silicon nitride attachment into a reaction sintered radome for even more strength in the attachment. The reason that this technique is feasible is that there is no dimensional change from the dry silicon shape to the reaction sintered shape, regardless of the density of the two pieces to be bonded. Therefore, two silicon pieces can be reaction sintered together, a silicon piece can be reaction sintered to a silicon nitride part, or two silicon nitride parts can be bonded together with silicon slip and a second reaction sintering.

2. Rain Erosion Sled Attachment

The Mauler radome prepared for sled testing uses an attachment design proven sled-worthy for slip-cast fused silica radomes tested at Mach 5 and should be satisfactory for use with the reaction sintered silicon nitride radome at Mach 4.

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V. CONCLUSIONS

1. Small variations in the dry grinding of silicon and in its dispersal in water can produce large variations in slip rheology and in the subsequent slip-cast density and reaction sintered properties.

2. The addition of pigment grade magnetite ($FeO \cdot Fe_2O_3$) improves the rheology of the casting slips, however, the magnetite must be added at the time the silicon is dispersed in water.

3. Addition of magnetite to an already prepared slip results in a different rheological system, a higher viscosity with less solids content, and high probability of an inferior casting.

4. Increasing the viscosity of silicon slips by the addition of magnetite at the time the slip is formed can result in a lower density casting which is easier to nitride.

5. Additional work is required to tie down all parameters for repeatable reaction sinterings, however, it is practical to obtain repeatable radome properties with few losses if the same slip is used for each casting and cryogenic nitrogen is used in each reaction sintering.

6. Silicon radomes slip-cast from dry ground commercial silicon powders dispersed in water can be repeatably reaction sintered to produce transmission losses of only a few dB.

7. Unsealed radomes pick-up moisture which degrades electrical transmission.

8. Radomes can be effectively sealed with silicone resins, either impregnating or surface sealing type without degrading electrical transmission.

9. Different densities of silicon nitride can be bonded together by reaction sintering using silicon slip as the adhesive.

VI. RECOMMENDATIONS

It is recommended that further work be accomplished to firmly establish all parameters controlling the properties of slip-cast silicon radomes. Further work should be accomplished in methods of sealing reaction sintered silicon nitride radomes. Scale-up to Sparrow size and larger radome shapes should also be considered, but a reaction sintering facility of different design than used in this program would be necessary.

Attachment systems for flight radomes other than silicon nitride should also be considered. One potential candidate for attachments is carbon composites. These can be tailored to adjust thermal expansion to match the reaction sintered silicon nitride 7/.

The ability to sinter together silicon nitride components of different density make the possibility of A-sandwich construction feasible. Research on methods of forming reaction sintered silicon nitride A-Sandwich radome structures should be conducted.

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4. Popper, P. and Ruddlesden, S. N., "The Preparation, Properties and Structure of Silicon Nitride," Trans. Brit. Ceram. Soc. 60, pp 603-626, September 1961.

5. Harris, J. N., "An Investigation of Reaction Sintered Silicon Nitride As A Radome Material," Final Report, Contract N00019-74-C-0248, Naval Air Systems Command (Code AIR-52032A), February 1975.

6. Private Communication with George Schmitt, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

7. Private Communication with Vance Chase, General Dynamics/San Diego.

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APPENDIX

TRANSMISSION CURVES FOR MAULER RADOMES

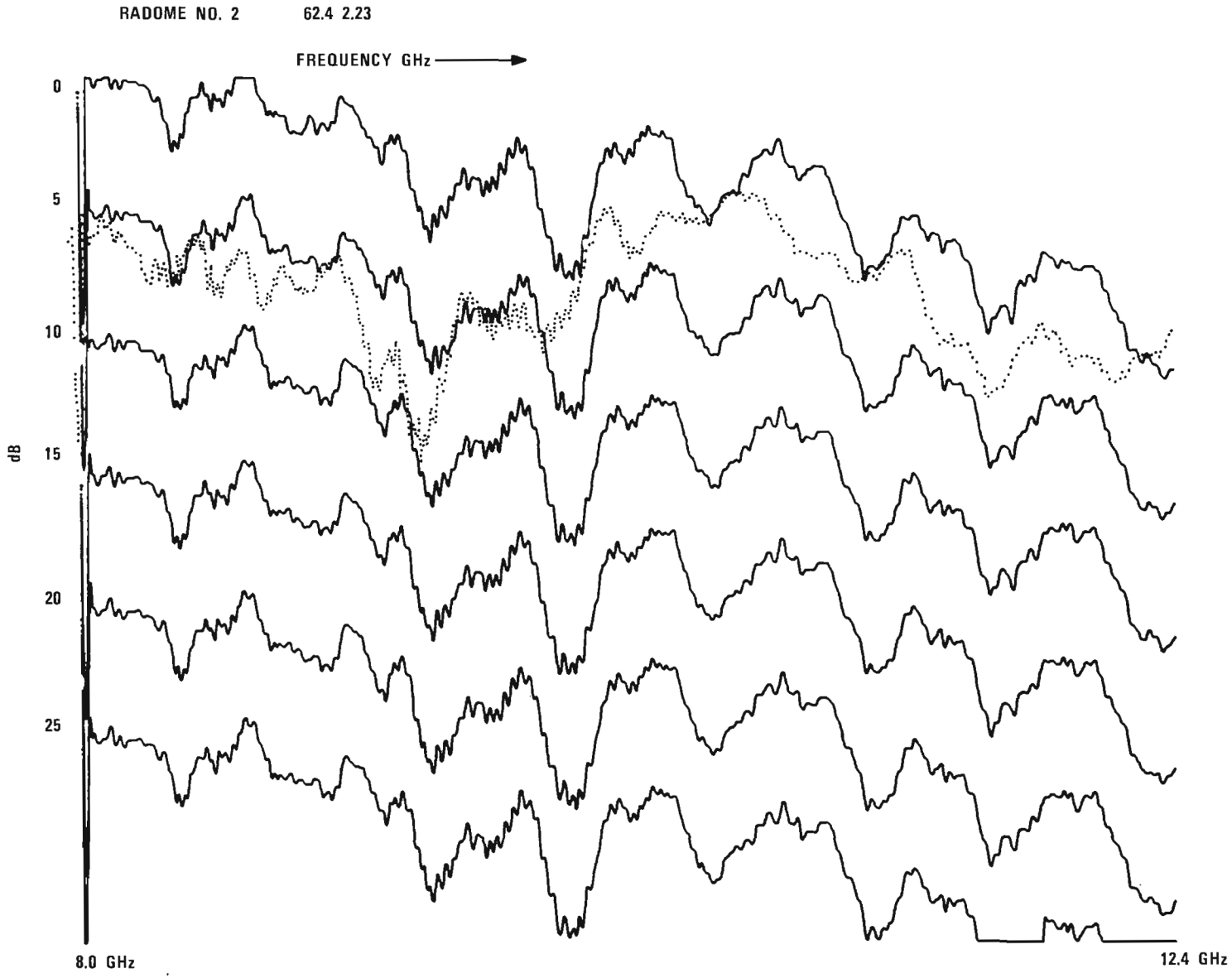

Figure 8. Transmission Through Radome Number 2 (After Thorough Drying).

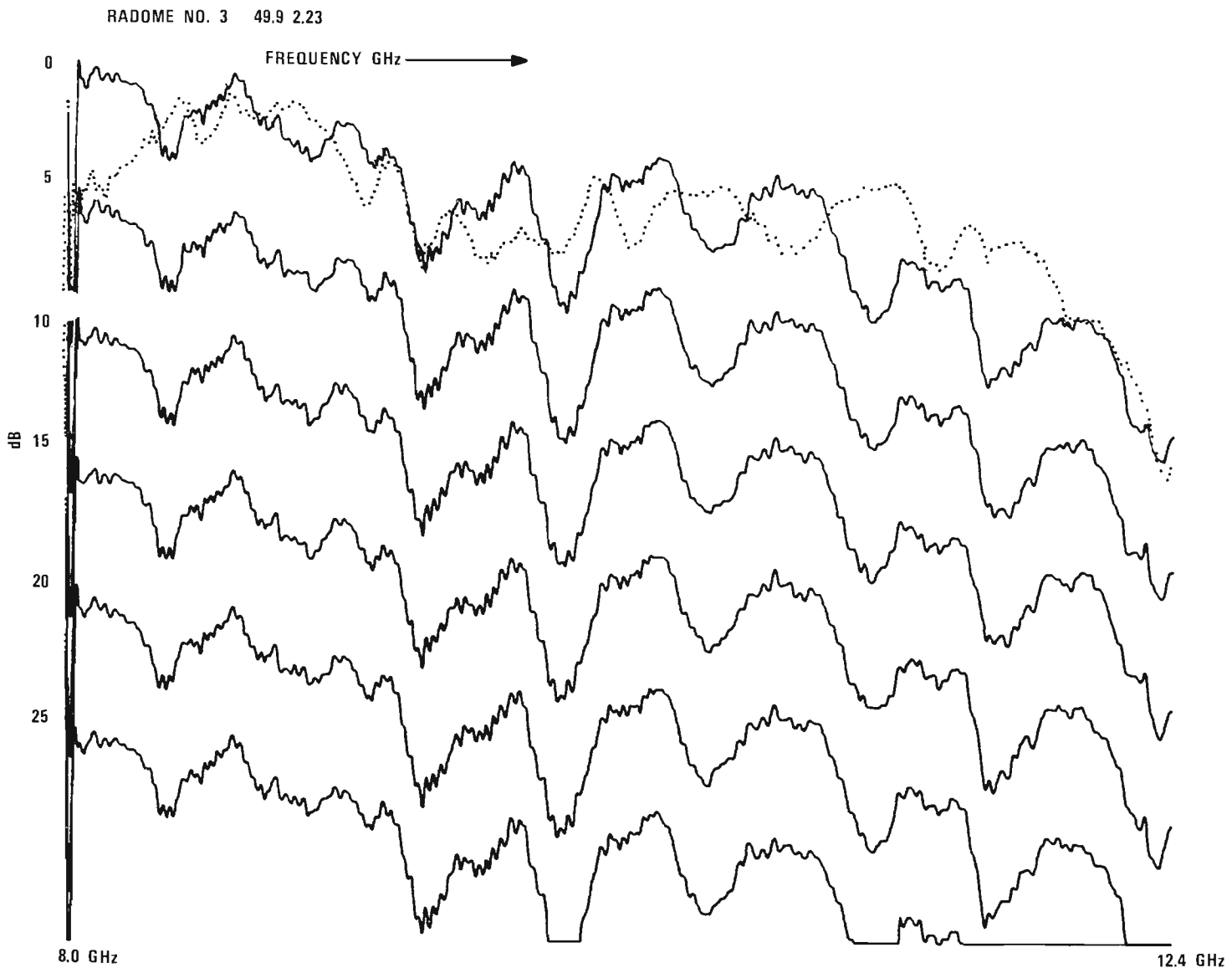

Figure 9. Transmission Through Radome Number 3 (After Thorough Drying).

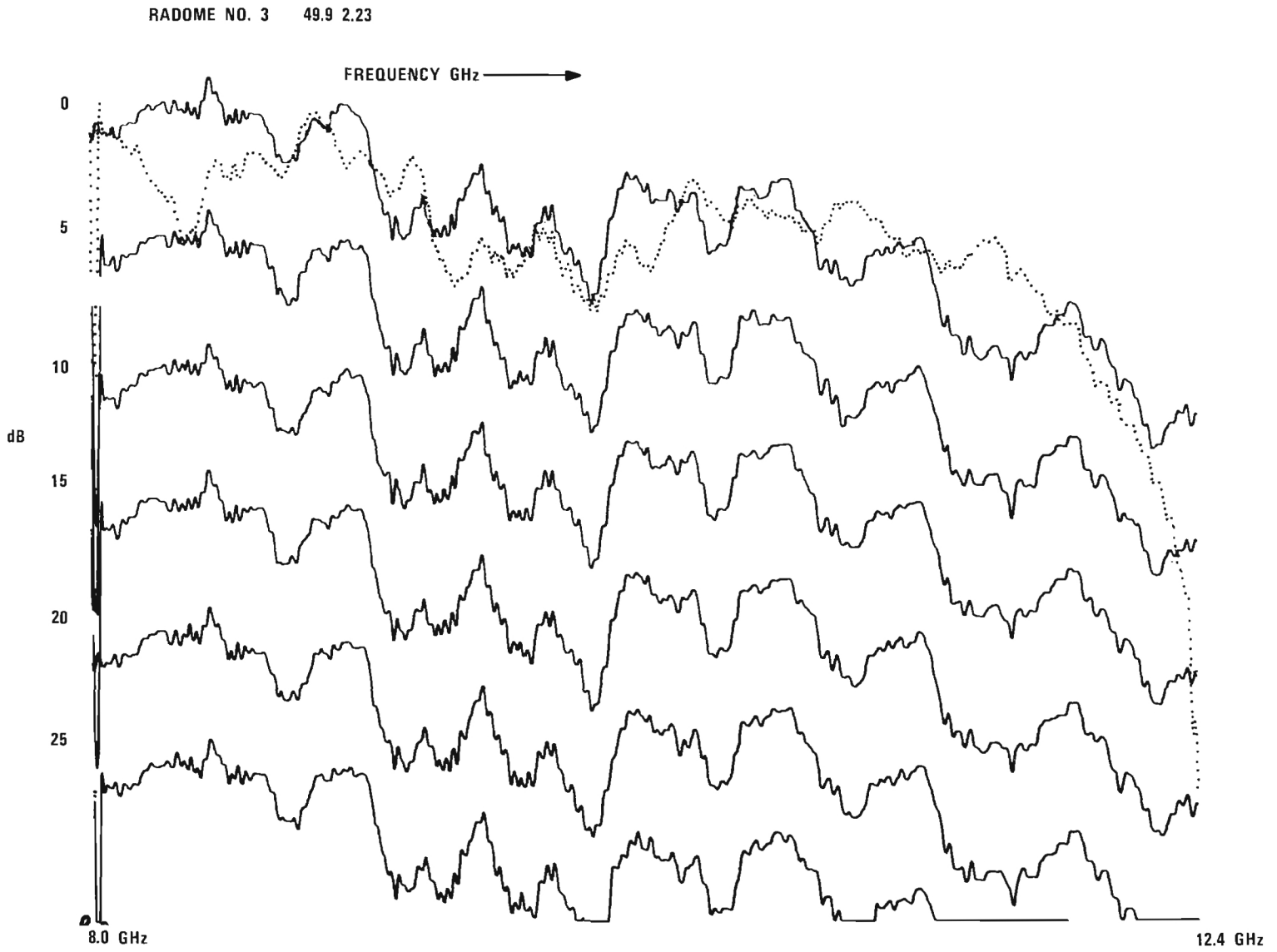

Figure 10. Transmission Through Radome Number 3 (After Exposure to Laboratory Humidity).

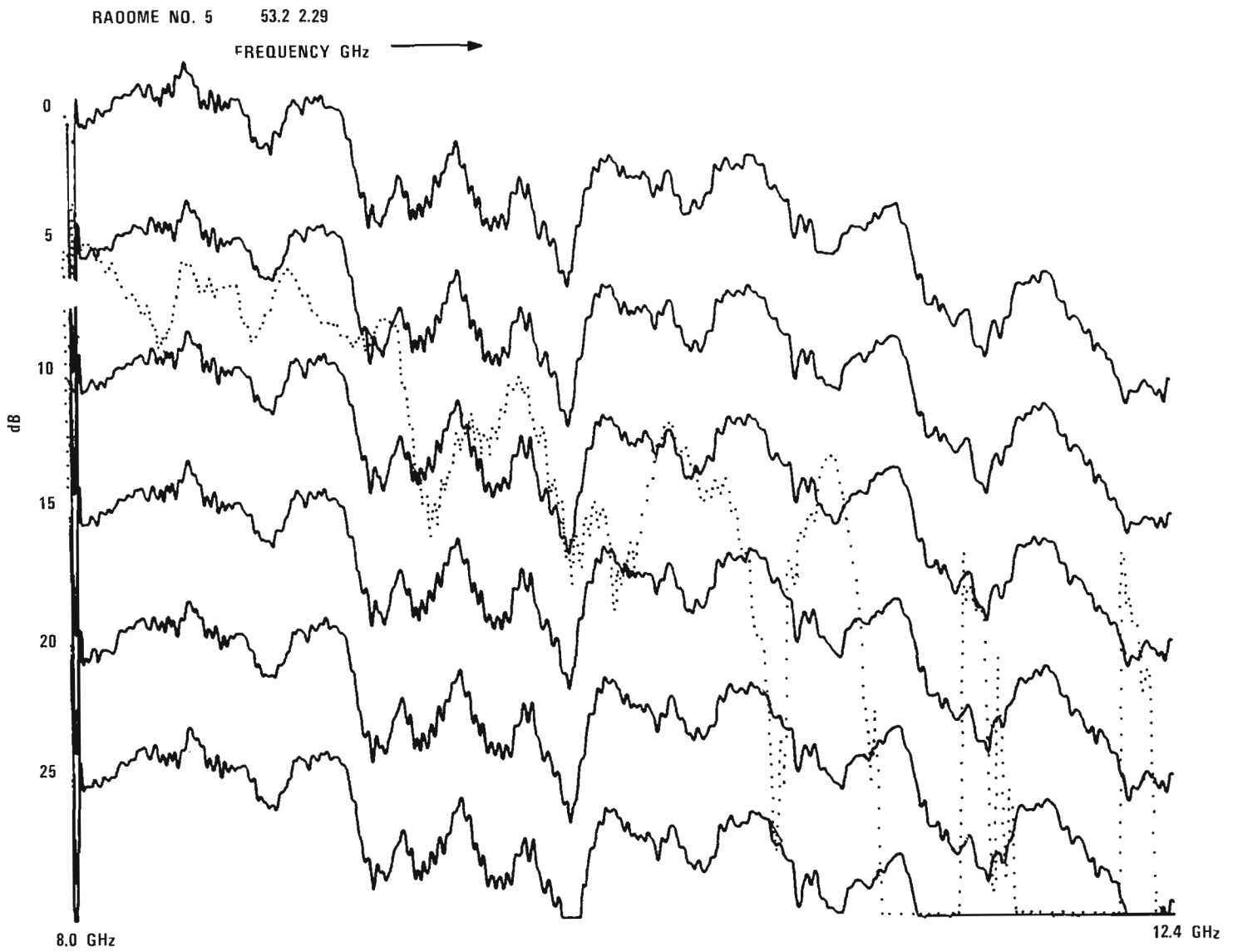

Figure 11. Transmission Through Radome Number 5 (After thorough Drying).

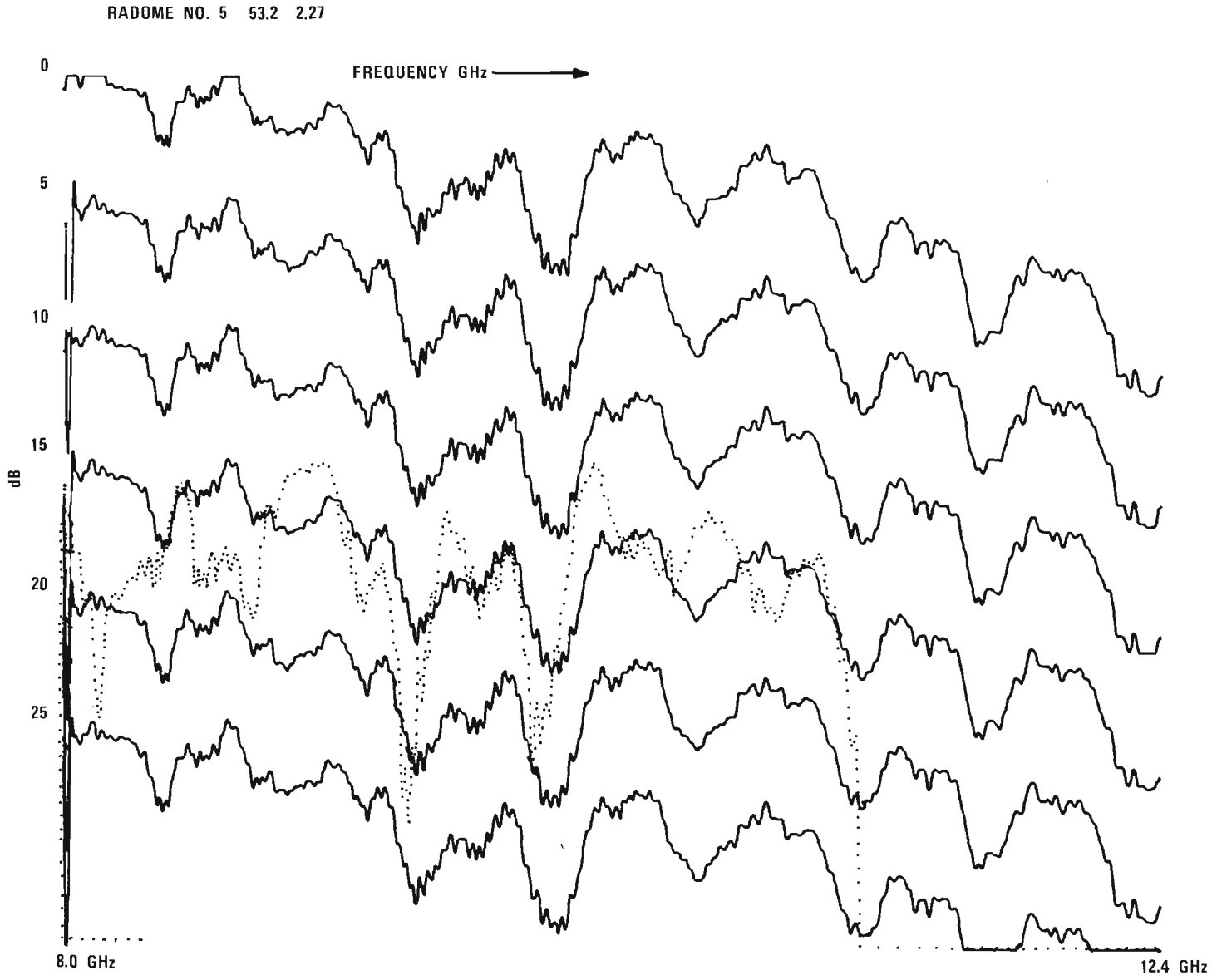

Figure 12. Transmission Through Radome Number 5 (After Exposure to Laboratory Humidity).

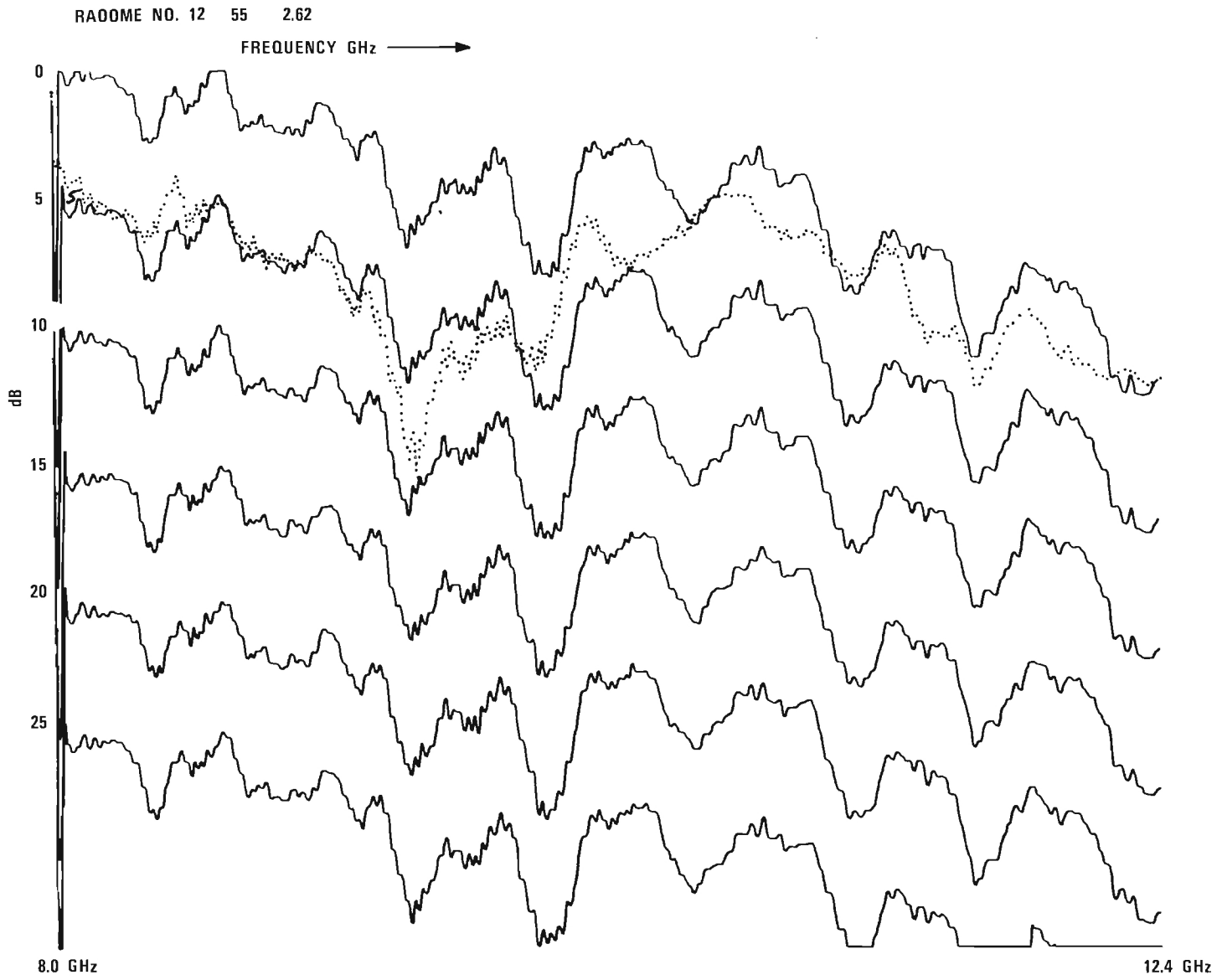

Figure 13. Transmission Through Radome Number 12 (After Thorough Drying).

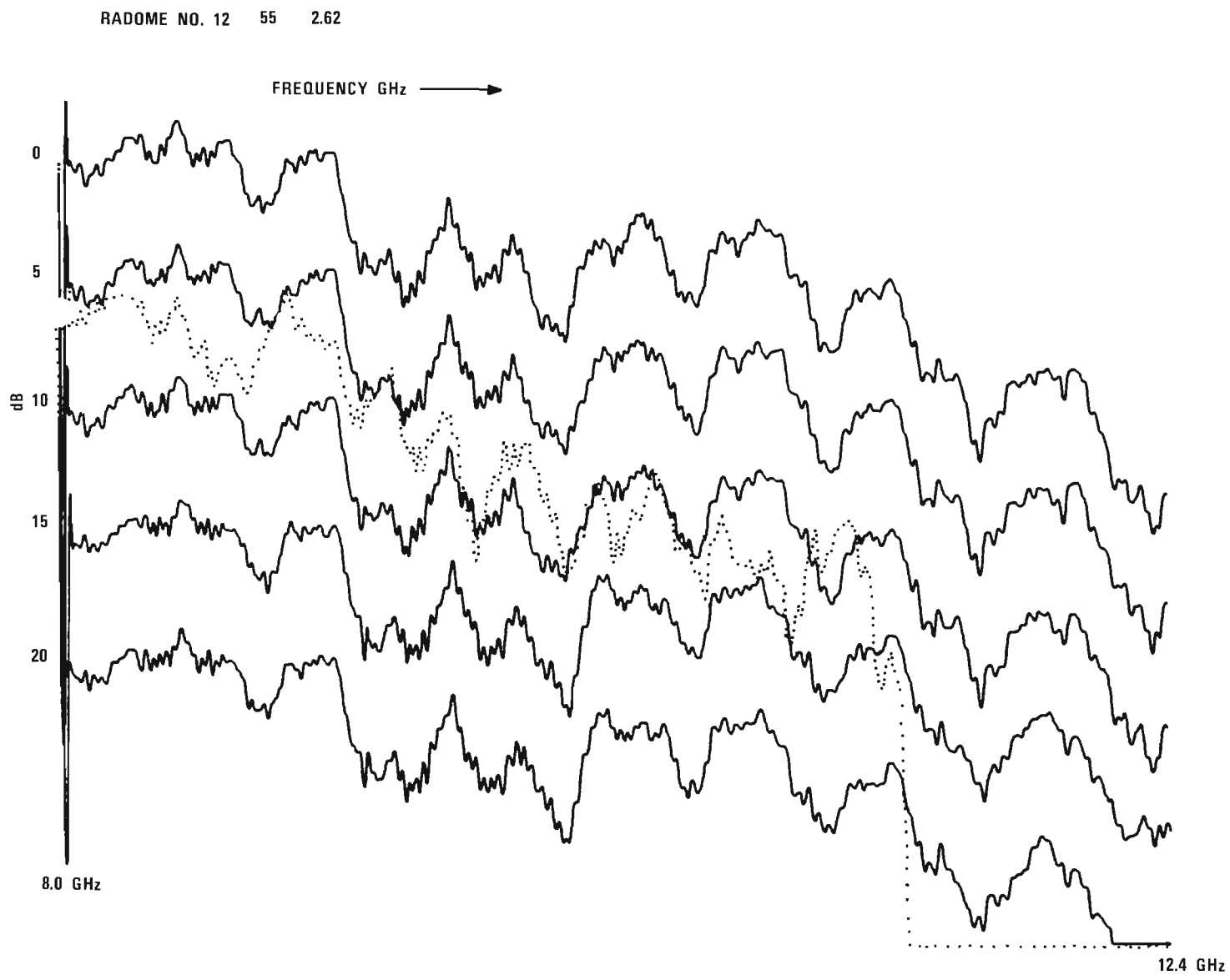

Figure 14. Transmission Through Radome Number 12 (After Exposure to Laboratory Humidity).

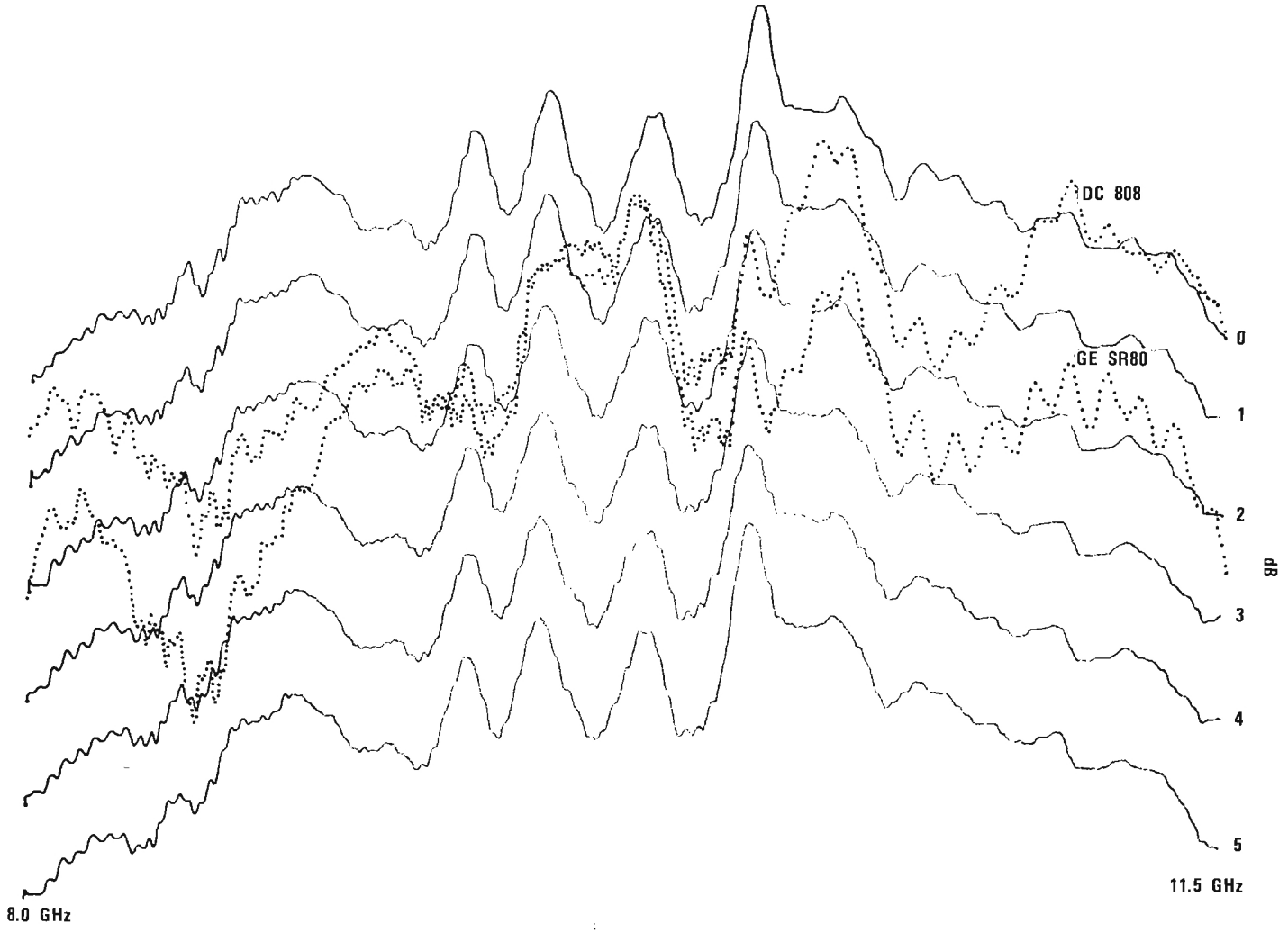

Figure 15. Transmission Through Radomes Sealed with Dow Corning 808 and GE SR-80 Resins.

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	6. PERFORMING ORG. REPORT NUMBER: A-1724
7. AUTHOR(s): Joe N. Harris	8. CONTRACT OR GRANT NUMBER(s): N00019-75-C-0372
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Engineering Experiment Station, Georgia Institute of Technology, Atlanta, Georgia 30332	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS: Naval Air Systems Command (Code AIR-52032-A) Department of the Navy	12. REPORT DATE: October 1975
	13. NUMBER OF PAGES: 51
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	15. SECURITY CLASS. (of this report): Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

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Reaction Sintered Silicon Nitride:
- Slip Casting
- Fabrication
- Reaction Sintering

Radomes:
- Electrical Properties

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Eleven reaction sintered silicon nitride radomes were prepared by slip casting silicon blanks and reaction sintering in nitrogen to; determine the repeatibility of electrical transmission characteristics, examine methods of sealing porous radomes, investigate possible flight attachment systems, and prepare a radome with attachment for rain erosion sled testing. Only two of fifteen castings were lost in the mold. Dry density varied from 1.58 to 1.89 gm/cm^3 due to variation in viscosity and particle size distribution of the slip. Final densities of radomes completing the

reaction sintering cycle varied from 2.55 to 2.76 gm/cm^3. Electrical transmission patterns were made on six of the radomes using swept frequency techniques. At the frequency representing a half-wave wall thickness for the particular density of each radome, transmission losses were of the order of a few dB. Weight gained during nitriding was not a good indicator of electrical transmission capability. Acceptable transmission patterns were made on radomes with apparent weight gains as low as 50 percent. Radomes sealed against moisture by impregnating or surface coating with silicone resin were not degraded in electrical performance by the sealing. A dense reaction sintered silicon nitride attachment ring was self-bonded into a lower density reaction sintered silicon nitride radome as a demonstration of the feasibility of this approach for attachment systems. Low expansion carbon composite rings tailored to match the expansion of silicon nitride were also considered for attachment rings. A 36 percent nickel low expansion steel bonded with an epoxy adhesive will be used in an attachment for rain erosion sled testing of a reaction sintered silicon nitride radome at a future date.

B.C.B.